QUALITY ASSURANCE PROJECT PLAN

WINDSOR & CADY BROOKS SEDIMENT & TURBIDITY MANAGEMENT FOR CLEVELAND RESERVOIR

March 2018

Massachusetts Department of Environmental Protection (DEP)
Water Quality Management Planning Grant Program:
Watershed Protection of Windsor & Cady Brooks (Project 055-1014-04 ARRA)

Grant Recipient:
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Pittsfield, MA 01201

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QUALITY ASSURANCE PROJECT PLAN

WINDSOR & CADDY BROOKS SEDIMENT & TURBIDITY MANAGEMENT FOR CLEVELAND RESERVOIR

March 2010
Massachusetts Department of Environmental Protection 604(b) Water Quality Management Planning Grant Program:
Water Quality Assessment of Windsor & Cady Brooks (Project #09-10/604 ARRA)

Grant Recipient: City of Pittsfield
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1.3 Distribution List

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11. Comprehensive Environmental Inc. Staff  
Field Sampling Team (Comprehensive Environmental Inc. and Berkshire Regional Planning Commission Staff)

A project personnel sign-off sheet is included in Appendix A and will be completed by the field sampling team prior to each sampling event since field teams may vary based on the availability of project personnel. This will allow the field team leader and project manager to identify sampling teams, if needed, to assist with data review, validation and verification procedures.
1.4 Project / Task Organization

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Field Team
CEI Staff
BRPC Staff

Laboratory QA Officer
Mark Perkins
781-848-7844
Project Manager
Ms. Melissa Provencher is the project manager. She serves as the Berkshire Regional Planning Commission’s (BRPC) primary contact and project manager for the Water Quality Assessment of Windsor & Cady Brooks Project, which includes this QAPP. Ms. Provencher is responsible for overall management of the project and will coordinate and oversee each phase with appropriate project team personnel.

MassDEP Project Contact
Mr. Gary Gonyea is the MassDEP Project Contact. Mr. Gonyea is responsible for overall MA DEP project grant management including technical and administrative supervision and final task review and approval.

MassDEP QA Officer
Mr. Richard Chase is the MassDEP Quality Assurance Officer. Mr. Chase will review and provide comment on the Quality Assurance Project Plan (QAPP). Modifications to the approved QAPP will be documented and submitted to Mr. Chase for approval, including but not limited to changes in sample design, sample collection, and data assessment and reporting.

U.S. EPA Project Officer
Ms. Mary Jo Feuerbach is the EPA Project Officer. Ms. Feuerbach is responsible for overall EPA grant management.

U.S. EPA QA Officer
Mr. John Smaldone is the EPA Quality Assurance Officer. Mr. Smaldone will review and provide comment on the Quality Assurance Project Plan (QAPP). Modifications to the approved QAPP will be documented and submitted to Mr. Smaldone for approval, including but not limited to changes in sample design, sample collection, and data assessment and reporting.

Project Manager & Technical Advisor
The BRPC has enlisted Comprehensive Environmental Inc. (CEI) to assist with project development, project management, budget, and technical oversight of the project tasks. Mr. David Nyman will serve as CEI’s project manager for the grant as well as serve in a technical advisory role. CEI will also be responsible for:
1. Turbidity monitoring at aqueduct inlets and at the Cleveland Reservoir treatment plant
2. Sediment sampling and bathymetric mapping at the Windsor and Cady Brook impoundments
3. Sediment yield modeling for the Windsor and Cady Brook watersheds

QA/QC Officer and Technical Advisor
Ms. Julia Capurso is the author of this QAPP and is responsible for QAPP revisions and submittals based on comments from the Project and QA Officers. She is the point of contact for QAPP inquiries and will also serve as the Technical Advisor for the QAPP with oversight from the Project Manager and QA/QC Officer. Ms. Capurso will use the QA/QC and technical
components in her role as the Field Team Leader as needed throughout the sampling project. She will ensure that the elements of the project follow the QA procedures in the QAPP including data quality objectives and reporting.

**Field Team Leader/Monitoring Coordinator**

CEI’s project manager will designate Mr. Nick Cristofori to serve as the Field Team Leader and Monitoring Coordinator. Mr. Cristofori will be responsible for coordinating the elements of field monitoring, provide assistance to CEI and BRPC staff and help the QA/QC Officer assess field monitoring performance.

Mr. Cristofori has been involved in the monitoring and water quality sampling of several other Towns within Massachusetts. He will hold a field start-up meeting for CEI staff that will be involved in sampling to review procedures, project goals and objectives and answer questions regarding the QAPP. Training will include sample collection and documentation, QAPP review, and a dry-run of the sampling event. The QAPP and other sampling references will be available prior to and during sampling events.

**Laboratory QA/QC Officer – Water Chemistry**

Mr. Mark Perkins is the Laboratory QA/QC Officer for Geolabs and will be responsible for laboratory QA/QC to ensure that the laboratory data quality objectives are achieved. Mr. Perkins will also coordinate lab schedules with the Field Team Leader / Monitoring Coordinator.

**Field Team**

The project Field Team will consist of members of both CEI’s and BRPC’s staff. The Field Team will consist of experienced field personnel.
1.5 Problem Definition / Background

Significant sediment deposits and the re-suspension of these materials during storm events or high flows may be contributing to increased turbidity impacting the Cleveland Reservoir, the City of Pittsfield's principal water supply. This project will assess sources of sediment to Windsor and Cady Brooks and identify appropriate management measures to prevent sediment from entering Cleveland Reservoir. Cleveland Reservoir is a Class A waterbody and supplies approximately 74% of Pittsfield's water. The Cleveland Reservoir Watershed is located in the towns of Hinsdale, Windsor, and Peru. Flow from Windsor and Cady Brooks is diverted from Windsor Reservoir to Cleveland Reservoir by a system of aqueducts.

The City of Pittsfield owns and operates the Cleveland Reservoir as its principle water supply with an approximate yield of 8 MGD. The water supply originates from the reservoir's relatively small (1.5 sq. mi.) immediate watershed and from the watersheds of two intake structures located on Windsor Brook and Cady Brook. Based on the available plans, the impoundments behind each diversion structure are less than one half acre in surface area. The watershed areas for the Windsor and Cady Brook diversions are approximately 9.3 and 3.2 square miles, respectively. The brooks naturally flow into Windsor Reservoir, which is a water supply source for the Dalton Fire District, but are redirected to the Cleveland Reservoir in connection with Pittsfield's water needs. A USGS site locus map showing the reservoirs and diversion structures is provided on Figure 1.5-1. Figure 1.5-2 shows the watersheds for Windsor and Cady Brook diversions. The diversion structures and impoundments for each brook are pictured in Appendix B.

These flow diversion structures contain significant accumulated sediment from the brooks. Past reservoir and watershed management activities have identified that sediment transported by these brooks, and associated turbidity under high flow and storm conditions, adversely impacts the quality of the Cleveland Reservoir water supply. Increased turbidity can lead to Water Department action to reduce flow withdrawals at the two intake structures. For example, the Water Department personnel begin to close the diversions when the turbidity of the brooks, combined with the flow from the Cleveland Reservoir, approaches 2 NTUs, which begins to impact the treatment capacity of the plant.

Significant sediment deposits are present at the Windsor and Cady Brook diversions and the re-suspension of these materials during storm events or high flows may be contributing to the increased turbidity that impacts Cleveland Reservoir. The City of Pittsfield intends to remove the accumulated sediment in the vicinity of the diversion impoundments consistent with previous maintenance activities at the Windsor Brook impoundment that removed -690 cubic yards of material in 1992.

As part of its comprehensive management approach to protect its water supply, the City of Pittsfield has initiated this study to review the characteristics of the watersheds of Windsor and Cady Brooks, characterize the conditions in these brooks that result in actionable levels of sediment.
turbidity at the Cleveland Reservoir intake structure, and develop an understanding of the sources of sediment from the tributary watersheds. The City will also collect data on bathymetry, accumulated sediment depth, and sediment physical and chemical composition, to assist in developing a future maintenance project for removing this sediment from the diversion structure impoundments.

1.6 Project/Task Description

The objectives of the Windsor & Cady Brook Sediment & Turbidity Management for Cleveland Reservoir are:

1. To attain a greater understanding of known and suspected contributors of sediment loads through data review, watershed reconnaissance, stream channel evaluation, turbidity monitoring, and modeling.

2. To evaluate the potential improvements through analysis of best management practice alternatives in the watersheds.

3. To identify conceptual solutions and recommendations for structural and/or non-structural best management practices that can be implemented to address priority problems identified.

In conducting this assessment, examples of questions that will be considered include:

1. What is the source of the sediment in each of these streams? Does it derive from land use and land disturbance activities in the contributing watersheds, does it derive from streambank erosion, or is it a combination of these sources, and to what extent are these sources attributable to human activities?

2. How is the turbidity experienced at the water intake structures affected by flows? Is there a difference in turbidity levels between Cady and Windsor Brook during storm events? What turbidity levels at the intake affect turbidity levels in the Cleveland Reservoir?

3. How can the sediment conveyed in these streams be reduced through source control practices or structural Best Management Practices (BMPs)?

4. In addition to mitigation of sediment loads, what other measures might effectively address the turbidity conditions encountered in the operation of the intake structures during storm events?

The purpose of the project, as described in the Project Scope of Work, is to address these questions through an initial analysis of the watershed, stream system, sediment characteristics, and potential management alternatives. Through the project, the baseline information and conceptual alternatives will be developed to form the framework for future planning and implementation that will protect the water quality of the subject streams and the water supplies that depend on them.

The following is a description of tasks included in the project and associated objectives. Appendix C outlines the project schedule.
1. **Quality Assurance Project Plan (QAPP)**

A QAPP will be developed in accordance with EPA guidance which will detail protocols for water quality-related monitoring and modeling. The QAPP will establish protocols for:

1. Modeling estimated generation of sediment based on GIS soils, stream network, and land use databases
2. Turbidity monitoring at the inlets to the aqueduct and at the Cleveland Reservoir treatment plant
3. Sediment bathymetry and sampling at the Windsor and Cady Brook impoundments.

2. **Characterization of Turbidity in Windsor and Cady Brook Impoundments**

Monitoring will be performed in accordance with the DEP/EPA approved QAPP developed under Task 1 to obtain information on the turbidity in the diversion structure impoundments at Windsor and Cady Brooks during varying flow conditions. The existing sediments that have accumulated in the impoundments will be quantified and characterized.

   a. **Impoundment / Aqueduct Turbidity Monitoring**

   Monitoring stations will be established at the aqueduct inlets for the Windsor and Cady Brook diversion structures to monitor turbidity over a designated four-month study period, to obtain information on how turbidity varies with flow. The monitoring stations will record stage data, which will be used to calculate flows at each structure by relating the observed stage to the flow-defining characteristics of the spillway and diversion weir. The observed relationship between turbidity and flow will be compared to the occurrence of elevated turbidity events at the Cleveland Brook reservoir water treatment plant, to ascertain how the turbidity levels of each of these two brooks relate to the operation of the plant.

   b. **Impoundment Bathymetry and Sediment Sampling**

   The Windsor and Cady Brook impoundments will be mapped to establish current bathymetry and sediment profiles. This information will be compared to 1948 design plans and the 1992 Intake Maintenance Plan for Windsor Brook, to develop an estimate the current sediment volume. This information will support a future maintenance project for the removal of sediment to restore impoundment capacity.

   Up to two samples of sediment will be obtained from each impoundment, and analyzed for chemical and physical properties. This information will also be used in support of future maintenance activity. The physical characterization will also provide an understanding of the particle sizes typically captured by the
3. Evaluation of Potential Sediment Sources

The Study Team will identify potential, significant sources of sediment to Windsor and Cady Brooks, based on available watershed data using GIS information, supplemented by a reconnaissance of the watershed, as described below.

a. GIS Map and Database

A GIS map and database will be compiled, including GIS layers indicating topographic elevations, soils, watersheds, stream systems, land use/cover, road networks, surficial geology and other pertinent information compiled from existing databases such as MassGIS and NEIWPCC files. The base mapping will be compared to aerial photographs and USGS maps at a scale of 1:24,000 to confirm accuracy. Printed maps at 1:24,000 scale will be used to verify that features significant to sources of sediment (e.g., type of land use/cover, road surface, stream flow) are consistent with information shown on the USGS maps (1988) while also reflecting recent changes or development in the watershed evidenced by aerial photographs (2005). As warranted by field observations, the GIS map will depict areas that may be particularly prone to erosion.

b. Watershed Reconnaissance

The Study Team will conduct a "windshield" reconnaissance of the watershed, including observations of existing land uses, paved and unpaved roadways, and representative stream segments. Observations from this reconnaissance will be used confirm land use data depicted in the GIS map file. This reconnaissance provides the opportunity to identify existing conditions that indicate individual sources of erosion, such as forestry activities, suburban development activities, agricultural practices, or mining activities. It will also include identification of potential locations for the application of structural Best Management Practices under Task 5.

4. Estimating Watershed Sediment Volumes

An estimate of the sediment yield over time will be developed from the land uses and streambank erosion in the Windsor and Cady Brook watersheds. This estimate will be used to evaluate the effectiveness of various BMPs to reduce sediment load.

a. Base Sediment Yield Analysis

The Study Team will develop a sediment yield model for each of the two major watersheds using the GIS database (as confirmed by reconnaissance activities),
and regional rainfall records. The model will be developed using the ArcView Generalized Watershed Loading Function (AVGWLF) model published by the Penn State Institutes of Energy and the Environment. The 2008 version of the modeling software has been developed and calibrated for application in the New England states and includes routines to model erosion from upland surfaces and lateral streambank erosion, to generate monthly volumes of sediment yield. The calibration of AVGWLF for New England states shows that reasonably accurate watershed sediment yields can be obtained for analyses on an average annual basis. An analysis period of ten years will be used to estimate average monthly and annual yields of sediment from each watershed under existing conditions. The model will also be used to estimate a base sediment yield for a hypothetical “natural forest” condition, to indicate the impact of human activity on the sediment generation in the watersheds.

For this study, the model will be used strictly as a computational tool, as the model employs the widely accepted Universal Soil Loss Equation, and provides a convenient method for estimating soil loss and sediment delivery by this standard procedure. To verify that model output lies within a reasonable range, separate hand calculations will be performed using similar methods. This procedure will provide a means to understand the relative impact of human activity in these watersheds, and an order-of-magnitude estimate of sediment volume anticipated at the diversion control structures. The estimate will be used to determine the dominant sources of sediment in the watershed and tailor a management approach by evaluating the appropriateness of specific practices relative to this estimate.

b. Intake Structure Impoundment Sediment Capacity Analysis

Using average sediment yields computed by the model, each intake structure impoundment will be evaluated for sediment trapping efficiency. Average sediment yields will be converted to average concentrations based on modeled flows to obtain base-load estimates of sediment trapping efficiency. Using published USDA soils descriptions, supplemented by sediment gradation results from the sediment sampling, a simple, theoretical sediment settling computation will be conducted for each impoundment to develop an order-of-magnitude estimate of the portion of sediment captured by the impoundment. This evaluation will enable an approximate assessment of the time period required to fill each impoundment with sediment. This analysis will help determine the role of the intake impoundments in future sediment management actions (i.e. if impoundments can serve as a sediment capture BMP) and establish maintenance schedules.

5. Evaluation of BMPs for Managing Sediment and Turbidity

The Study Team will identify a range of potential management actions and practices for reducing sediment loads to study area streams. The team will also evaluate management
practices relative to the feasibility of implementation, cost, and effectiveness at removing sediment at the downstream end of each watershed. Potential management measures will be developed for future implementation to control and manage sediment inputs.


A tentative list of management actions will be developed to address sediment and turbidity based on the information and findings developed through previous tasks. Potential BMPs may include: structural BMPs, stream channel stabilization measures, operation and maintenance practices, and non-structural best management practices.


If broad-based management actions affecting large areas appear warranted by the initial BMP analysis, then the AVGWLF model would be revised and run for such a scenario (e.g., application of a specific agricultural practice to a sizeable portion of the watershed). For some agricultural management practices, an associated model (the PRedICT model) may be used to assess various scenarios of management actions). However, these models have a limited suite of BMP practices and thus have limitations in the evaluation of small, localized BMPs. Therefore, future scenarios involving BMPs outside the scope of these models will be analyzed using simple hand or spreadsheet calculations. For example, a reduction in road sand application in tons per year can be computed, and compared to the baseline sediment load estimated for existing conditions using the model. As noted above, the intent is to use the model as a convenient computational tool, which can then provide an order-of-magnitude baseline estimate of the amount of sediment anticipated from the study watershed.

Up to three scenarios will be evaluated using the appropriate computational methods, to assess the effectiveness of implementing various management actions.

c. Recommended Action Plan

The Study Team will develop a recommended action plan for sediment management based on the analysis conducted under Task 5B. The top five most cost-effective BMPs will be advanced to conceptual design. The action plan will set forth a list of action items recommended for implementation under future projects, including structural, non-structural, and operational components to reduce sediment load or alternatively to address remaining sediment load and turbidity issues.

6. Reporting

Monthly and quarterly progress reports as well as draft/final study reports will be
submitted to the DEP 604b Project Officer in accordance with the milestone schedule and 604b reporting requirements.
1.7 Data Quality Objectives for Measurement Data

The objective of this project is to collect sufficient data to determine an appropriate and effective approach to decrease or manage sediment loads in the Windsor and Cady Brook watersheds and/or diversion structure impoundments to maintain lower turbidity levels at the Cleveland Reservoir water treatment plant.

Turbidity data collected at each diversion structure will be qualitatively compared with the occurrence of actionable turbidity levels at the Cleveland Brook Reservoir water treatment plant. This comparison will assist in determining whether there may be appropriate actions by treatment plant personnel to manage high turbidity events. For example:

- When the reservoir experiences actionable turbidity levels, typically 2 NTU at the raw water line, are one or both streams also experiencing elevated turbidity levels?
- Is there a time lag between high turbidity events in the streams and those in the reservoir? If so, would ongoing monitoring at each impoundment allow the treatment plant personnel to manage turbidity and the gate structures more proactively?
- Are high turbidity events associated with a threshold flow condition, or can these events occur during non-storm events as well?

Turbidity data will be collected in the field using the In-Situ Troll 9500 and at the Cleveland Reservoir Water Treatment Plant using a Hach 1720E Low Range Turbidimeter. Data quality objectives for these instruments is shown in Table 1.7-1 below.

<table>
<thead>
<tr>
<th>Source Matrix</th>
<th>Indicator</th>
<th>Units</th>
<th>Reporting Limit</th>
<th>Detection Limit</th>
<th>Accuracy / Bias</th>
<th>Overall Precision</th>
<th>Measurement Range</th>
</tr>
</thead>
<tbody>
<tr>
<td>Diversion Structure Inflow</td>
<td>Turbidity</td>
<td>NTU</td>
<td>N/A</td>
<td>2</td>
<td>±5% of reading or ±2, whichever is greater</td>
<td>2</td>
<td>0 to 2000</td>
</tr>
<tr>
<td>Treatment Plant Raw Water</td>
<td>Turbidity</td>
<td>NTU</td>
<td>(Actionable level of 2 NTU)</td>
<td>0.015</td>
<td>0 to 40: ±2% of reading or ±0.015, whichever is greater 40 to 100: ±5% of reading</td>
<td>0.015</td>
<td>0 to 100</td>
</tr>
<tr>
<td>Treatment Plant Raw Water (QC Check)</td>
<td>Turbidity</td>
<td>NTU</td>
<td>0.001 on lowest range</td>
<td>0 to 1000: ±1% of reading or ±0.01, whichever is greater 1000 to 4000: ±5% of reading</td>
<td>0.01</td>
<td>0 to 4000</td>
<td></td>
</tr>
<tr>
<td>Water Level</td>
<td>Elevation (Depth relative to elevation reference)</td>
<td>ft</td>
<td>N/A</td>
<td>0.018</td>
<td>±0.05%</td>
<td>0.018</td>
<td>0 to 35</td>
</tr>
</tbody>
</table>
Laboratory data obtained from the sediment sampling will be used to help characterize the sediment capture efficiency of the impoundments. Sediment sample characterization will also be used to support the City’s future maintenance activities (such as sediment removal to restore sediment capacity) at the impoundments. Sediments must be analyzed according to DEP standards for analyzing material proposed for dredging and disposal. Table 1.7-2 below outlines data quality objectives for each indicator to be analyzed and corresponding DEP standards for dredging and disposal.

Table 1.7-2. Laboratory Indicator Data Quality Objectives for Sediment Analyses

<table>
<thead>
<tr>
<th>Indicator</th>
<th>Units</th>
<th>Reporting Limit</th>
<th>Detection Limit</th>
<th>Accuracy / Bias</th>
<th>Overall Precision</th>
<th>Measurement Range</th>
<th>DEP Reporting Limit for Dredging</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sieve Analysis</td>
<td>%</td>
<td>0.1%</td>
<td>0.1%</td>
<td>N/A</td>
<td>20% RPD</td>
<td>0% to 100%</td>
<td>Sieve Nos. 4, 10, 40, 60, 200</td>
</tr>
<tr>
<td>TOC</td>
<td>mg/kg</td>
<td>400</td>
<td>100</td>
<td>80% to 120%</td>
<td>20% RPD</td>
<td>Dilution possible</td>
<td>0.1%</td>
</tr>
<tr>
<td>EPH</td>
<td>ug/kg</td>
<td>2.2</td>
<td>1.0</td>
<td>40% to 140%</td>
<td>50% RPD</td>
<td>25</td>
<td></td>
</tr>
<tr>
<td>Arsenic</td>
<td>mg/kg</td>
<td>5.0</td>
<td>1.0</td>
<td>85% to 115%</td>
<td>20% RPD</td>
<td>0.5</td>
<td></td>
</tr>
<tr>
<td>Cadmium</td>
<td>mg/kg</td>
<td>1.0</td>
<td>0.085</td>
<td>85% to 115%</td>
<td>20% RPD</td>
<td>0.1</td>
<td></td>
</tr>
<tr>
<td>Chromium</td>
<td>mg/kg</td>
<td>5.0</td>
<td>0.19</td>
<td>85% to 115%</td>
<td>20% RPD</td>
<td>1.0</td>
<td></td>
</tr>
<tr>
<td>Copper</td>
<td>mg/kg</td>
<td>5.0</td>
<td>0.187</td>
<td>85% to 115%</td>
<td>20% RPD</td>
<td>1.0</td>
<td></td>
</tr>
<tr>
<td>Lead</td>
<td>mg/kg</td>
<td>5.0</td>
<td>0.44</td>
<td>85% to 115%</td>
<td>20% RPD</td>
<td>1.0</td>
<td></td>
</tr>
<tr>
<td>Nickel</td>
<td>mg/kg</td>
<td>5.0</td>
<td>0.102</td>
<td>85% to 115%</td>
<td>20% RPD</td>
<td>1.0</td>
<td></td>
</tr>
<tr>
<td>Zinc</td>
<td>mg/kg</td>
<td>5.0</td>
<td>0.64</td>
<td>85% to 115%</td>
<td>20% RPD</td>
<td>1.0</td>
<td></td>
</tr>
<tr>
<td>Mercury</td>
<td>mg/kg</td>
<td>0.25</td>
<td>0.02</td>
<td>85% to 115%</td>
<td>20% RPD</td>
<td>Dilution possible</td>
<td>0.02</td>
</tr>
</tbody>
</table>

TOC: Total Organic Carbon  
EPH: Extractable Petroleum Hydrocarbons  
QCS: Quality Control Standards  
RPD: Relative Percent Difference
The bathymetric survey and associated sediment profiles will be used to estimate the volume of sediment deposited in each impoundment.

To formulate a general idea of annual sediment loads in each watershed, the AVGWLF model will be used as a computational tool to estimate the baseline volume on a monthly and annual basis.

Together, these pieces of information will be used to help identify management practices for controlling sediment inputs and or implementing actions in response to elevated turbidity levels in the two watersheds.

Data quality objectives will include precision, accuracy/bias, representativeness, comparability and completeness:

**Precision** is a measure of the reproducibility of repeated measurements. For the bathymetric mapping, precision will be verified by a repeated measurement built into the SOP contained in Appendix D. Depth to the surface of sediment will be first measured by secchi disk to ensure there is no penetration into soft sediments. The measurement will then be transferred to the rod used for the sediment thickness using a zip-tie as a marker. Before probing into the sediment, field staff will verify the water depth by lowering rod to surface of sediment and checking the zip-tie relative to water level. Due to field subjectivity of soft sediment depth measurements, the obtained data will be verified against available drawings of the original impoundment bathymetry.

Precision of turbidity data will be determined by calibration of the turbidity instrument by the procedure described in the SOP. Precision of water depths logged by the turbidimeter will be determined by verifying data with the installed staff gage; this procedure will occur three times in the first month of monitoring and subsequently once per month and during field activities where specified in the SOPs contained in Appendix D.

**Accuracy/Bias** is an indicator of measurement confidence and will be calculated based on analytical results of laboratory spiked samples of known chemical concentrations. Laboratory analysis will include the division of a sample into two aliquots with a known amount of a standard added to one aliquot. Both aliquots are then analyzed and the amount of the spiked material recovered will be compared to the amount added. Spike samples will be analyzed at a frequency of one per analytical batch. Laboratory spikes will not be performed for parameters measured by the turbidimeter due to the impracticality of this analysis. Turbidity measurements will be compared against the manufacturer’s calibration solution.

Percent recoveries are calculated as follows: 

\[
\%\text{Recovery} = \left(\frac{\text{SC} - \text{UC}}{\text{EV}}\right) \times 100
\]

Where:

- \(\text{SC}\) = Concentration in the spiked sample
UC = Concentration in the unspiked sample
EV = Expected value

**Representativeness** is the extent to which the sampling design and measurements obtained adequately reflect the true environmental conditions at the location being monitored. The selection of monitoring locations was based upon knowledge that the Windsor and Cady Brook impoundments have been historically problematic for the Cleveland Reservoir treatment plant. One purpose of the turbidity monitoring at each impoundment is to determine whether the conditions in the impoundments represent or correlate to turbidity issues observed at the treatment plant. The measurements obtained in the field, therefore, will indicate whether there is a correlation between water quality at the selected locations and water quality at the overall location being considered.

Representativeness of sediment sampling will be achieved by selecting locations on sediment deposits with a wide gradation of materials (i.e., on the downstream end of a sediment point bar). The samples will also be taken from six to eight inches below the surface and collected from a composite of core samples at various depths.

**Comparability** is the degree to which data can be compared directly to similar studies. Data obtained during the sediment profiles and bathymetry will be compared to original design drawings of each impoundment. The sediment depths estimated from the field measurements will be verified by determining the original depth of the impoundment. With the longer-term nature of the turbidity data, analysis of downloaded data will include a comparison to previously collected data to qualitatively determine whether current data reflects patterns also shown in past data. Comparability of this data set to turbidity measurements at the water treatment plant will be enhanced by analyzing the instrument calibration solutions using the spectrophotometer at the plant. This spectrophotometer is used to analyze grab samples of raw water as a quality control check on the in-line Hach turbidimeter. A reading will be taken from the calibrated spectrophotometer for a known calibration solution used for the In-Situ Troll 9500. The measurement will be used as the new calibration value, should the observed versus “known” values differ, to establish a ground of comparison between field data and water treatment plant data.

Comparing between data from each impoundment will help determine appropriate sediment management strategies for the future. The turbidity data will help determine if long-term monitoring is a viable and appropriate management tool to use real-time readings at each impoundment to proactively take action to prevent high turbidity levels at the treatment plant.

**Completeness** is the percentage of validated data compared to the total amount of data collected. The data collected will be considered complete when 90% of it meets the above established criteria (precision, accuracy/bias, representativeness, and comparability).

Completeness may be calculated as follows:
Completeness = \( \frac{A}{B} \times 100 \)

Where:
A = Total number of valid data points
B = Total number of data points
1.8 Training Requirements / Certification

Staff from CEI and BRPC will complete the project monitoring and sampling. The Field Team Leader / QA/QC Officer will meet with the Project Manager and Technical Advisor to review the sampling sites, sampling procedures, project goals and objectives and to answer questions regarding the QAPP.

The Field Team Leader will visit each sampling location prior to holding the monitoring/sampling start-up meeting for the sampling staff. He will train the sampling staff, as appropriate for the sampling project based on prior sampling experience. At a minimum, training will include sediment sample collection and documentation, instrument installation and configuration, staff gauge installation, QAPP review, and a dry run of the sampling event.

The Field Team Leader and other staff from CEI to be paired with staff from BRPC for field activities have completed 40-hour OSHA HAZWOPER training and 10-hour Construction Outreach training. These certifications include field safety practices relevant to the planned field activities. CEI staff have also completed in-house training in field sampling methods, data collection and surveying.

The Project Manager and Technical Advisor is an environmental engineer with over 35 years of experience, including substantial work in regarding issues in stream systems, watershed management and stormwater management. He holds substantial expertise in assessments using the fundamental methods upon which AVGWLF was constructed. The additional technical staff contributing to this aspect of the project offers three years of experience including previous implementation of the non-spatial GWLF model, work in spatial analysis, and implementation of AVGWLF in New England for the purpose of surface water supply watershed management.

The QAPP and other sampling references will be available prior to and during sampling events. Most of the staff is already trained in sampling and data collection techniques. Appendix A includes a copy of the training record that will be completed and maintained on file with the City of Pittsfield and included as part of the final project report.
1.9 Documentation and Records

Documentation accompanying samples from field collection and monitoring through laboratory analysis include field book records, sample labeling, chain of custody forms and lab data sheets.

Field Data Records

Field data will be recorded on-site at the time of sampling and data collection. Field books will be provided to the field team. Field records for bathymetric mapping, sediment profiles and sediment sampling will include the following information:

- Site name and location
- Names of field team personnel
- Data or sample date/time collection
- Data or sample collection point and reference locations (sketch of impoundment)
- Data or sample collection point and reference measurements (see Appendix D for Standard Operating Procedure)
- Weather conditions
- Site observations

Hard-copy records will be stored with CEI. The information will also be scanned into Adobe Reader format (.pdf files) and stored electronically on servers at CEI for a minimum of five years and backed up several times each week.

Sample Labeling

Sample labeling will include a label for each sample jar collected in the field and filled out with an indelible writing instrument. A sample label is included in Appendix E and will include the following information:

- Name of sample collected
- Sample identification
- Location of sample
- Date of collection
- Time of collection
- Initials of person collecting the sample
- Preservative
- Analysis

Records will be maintained at Geolabs in an archive for ten years.

Chain of Custody

A chain of custody will be used to track the samples from the collection point through analysis.
and data management. Chain of Custody records will be signed by the appropriate Field Sampling Team member before samples are relinquished to the laboratory. The records will also be signed by the receiving laboratory. The Field Team Leader and the laboratory analyzing the sample will maintain copies of the Chain of Custody. Appendix E contains the appropriate sample Chain of Custody form. The Chain of Custody will contain the following information:

- General project information
- Sample identification
- Date of collection
- Time of collection
- Sample matrix
- Sample analysis
- Additional comments as appropriate

Records will be maintained at Geolabs in a central lab archival location for several years. Paper copies will be scanned into Adobe Reader format (.pdf files) and stored electronically on servers at CEI for a minimum of five years and backed up several times each week.

**Lab Data Sheets**
Lab data sheets will be used by the laboratory to document sample processing and analysis. Lab data sheets will be kept for each parameter analyzed and will include the following information:

- Name of lab
- Analysis date
- Time of sample arrival at lab
- Time samples were analyzed
- Sample ID # and site #
- Raw results, calculated results
- Name of lab analyst
- Internal QC procedures

Records will be maintained at Geolabs laboratories in a central lab archival location for several years. Paper copies will be scanned into Adobe Reader format (.pdf files) and stored electronically on servers at CEI for a minimum of five years and backed up several times each week.

**Data Presentation**
Data will be tabulated into various tables and figures and presented in the draft and final reports to be submitted to EPA and MA DEP for approval. Reports will include both a summary of data as well as copies of the raw data obtained during sampling and data collection events. Summaries will be presented in tables and figures within the report body with raw data included in an appendix. Monthly and quarterly reports will document data obtained in the most recent sampling round.
Modeling Documentation
Documentation of modeling efforts will include record of default input data, changes made to these inputs based on field observation, trials and results. Narrative explanation of these records, assumptions, and interpretation of results will also be included.

Report Presentation
A final report including the information listed in Section 3.2 will be compiled to document the overall work and outcome of the project.

Approved QAPP
Upon approval by MA DEP and EPA, the approved QAPP will be distributed to those specified on the Distribution List as outlined in Section 1.3. Distribution will be done via email, with hardcopies available to CEI staff and the Field Team prior to the first sampling event. Updates will be sent via email.
2.0 Data Generation and Acquisition

2.1 Data Collection and Sampling Process Design

The goal of this project is to collect data that will reflect the sources and relative volumes of sediment deposits and re-suspended sediments moving through the Windsor and Cady Brook diversion structures towards Cleveland Reservoir. This information will allow the project team to identify appropriate management measures to mitigate turbidity problems at the Cleveland Reservoir water treatment plant. Currently, management actions to mitigate these issues are taken when the turbidity of raw water coming into the treatment plant reaches a level of approximately 2 NTU, when treatment plant staff visit the diversion structures are closed, instead directing full stream flows to Windsor Reservoir.

The data to be collected for this project will include field turbidity monitoring at each impoundment, existing and on-going turbidity and precipitation data collected at the treatment plant, sediment sampling in the impoundments, and a bathymetric survey of each impoundment including sediment profiles. Turbidity monitoring, sample collection, bathymetric mapping and sediment profiles will be completed according to the respective SOPs located in Appendix D.

Turbidity Monitoring
Monitoring stations will be established at the aqueduct inlets for the Windsor and Cady Brook diversion structures. Selection of these locations was based on historical sediment issues that proved problematic for the Cleveland Reservoir water treatment plant. Monitoring data collected at these sites (by automated instruments at the two diversion structures) will be used to evaluate the type (e.g., thunderstorms) and frequency of flow events that are likely to generate the elevated turbidity that prompts the Pittsfield Water Department to begin closing the aqueduct intakes. The In-Situ Troll 9500 will be installed on-site at each diversion structure to automatically log turbidity and depth of water over a four-month monitoring period with an estimated data collection interval of 30 minutes.

Turbidity monitoring of raw water will be conducted at the Cleveland Reservoir treatment plant. The real-time turbidimeter will allow Pittsfield Water Department personnel to notify Monitoring Coordinator of instances where turbidity levels prompted an action (e.g., adjusting the flow through the diversion structures). Precipitation data will also be collected at the treatment plant.

Impoundment Sediment Sampling
The characterization of sediment in each impoundment will be completed with up to two sediment samples. These samples will be collected from existing sediment deposits in each impoundment by a qualified field team member. The sediment samples will be analyzed for the following parameters:
- sieve analysis with hydrometer
- total organic carbon
- extractable petroleum hydrocarbons
- heavy metals:
  - arsenic
  - cadmium
  - chromium
  - copper
  - lead
  - mercury
  - nickel
  - zinc

This will be used to evaluate the nature of sediments deposited in each impoundment and estimated sediment removal from impoundment maintenance. These analyses can be used to support a possible 401 Water Quality Certification for Dredging in the future.

Sample collection will be during daylight hours generally between 7 AM and 5 PM on a weekday(s). Samples will be brought back to the CEI office, where Geolabs will pick up and provide courier service to the lab within the appropriate holding time. The Field Team Leader and Monitoring Coordinator will coordinate the sample date and time with Geolabs in advance and immediately preceding the sampling event. Sediment samples will be composite samples collected using hand tools from land.

**Impoundment Bathymetric Survey and Sediment Profile**

A bathymetric map and sediment profile will be developed using hand tools to determine the depth of water and depth of sediment. Sediment thickness will be estimated by measuring the depth of water at the sediment surface and again at the firm underlying bottom below the sediment. This information will be used to estimate the sediment volume that has accumulated since the most recent maintenance activities and to understand the nature of the material that has been deposited. The bathymetry and sediment profile data will be collected from a canoe or comparable small water craft.
2.2 Data Collection and Sampling Method Requirements

Turbidity Monitoring
Data collected by the field turbidimeters will be downloaded monthly by a qualified field team member. Staff gages, which will be installed at each diversion structure, will be used to validate depth readings from the In-Situ Troll 9500 during monitoring events and data downloads. At times when the levels of turbidity observed at the Cleveland Reservoir water treatment plant prompt a management action, the Pittsfield Water Department will notify the Field Team Leader/Monitoring Coordinator. During these events, the Department will provide the turbidity measurement, the associated date and time of the measurement, and the action that was prompted.

Impoundment Sediment Sampling
The sediment samples will be collected from existing sediment bars using hand tools and a push-probe sampler. Sample collection methods are summarized in Table 2.1. Detailed laboratory QA/QC procedures are provided in Appendix F. Sample collection SOPs are provided in Appendix D.

Table 2.1 Container, Sample Size, Type, Preservation and Storage

<table>
<thead>
<tr>
<th>Indicator</th>
<th>Container Type</th>
<th>Minimum Sample Quantity</th>
<th>Sample Type</th>
<th>Preservation</th>
<th>Maximum Holding Time</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sieve Analysis with Hydrometer</td>
<td>Plastic Bag (Ziploc)</td>
<td>Quart (Ziploc)</td>
<td>Grab</td>
<td>NA</td>
<td>NA</td>
</tr>
<tr>
<td>Total Organic Carbon</td>
<td>Glass</td>
<td>4 oz</td>
<td>Grab</td>
<td>Ice (4°C)</td>
<td>14 days</td>
</tr>
<tr>
<td>Extractable Petroleum Hydrocarbons</td>
<td>Amber Glass</td>
<td>8 oz</td>
<td>Grab</td>
<td>Ice (4°C)</td>
<td>14 days</td>
</tr>
<tr>
<td>Arsenic</td>
<td>Glass</td>
<td>8 oz</td>
<td>Grab</td>
<td>Ice (4°C)</td>
<td>180 days</td>
</tr>
<tr>
<td>Cadmium</td>
<td>Glass</td>
<td>8 oz</td>
<td>Grab</td>
<td>Ice (4°C)</td>
<td>180 days</td>
</tr>
<tr>
<td>Chromium</td>
<td>Glass</td>
<td>8 oz</td>
<td>Grab</td>
<td>Ice (4°C)</td>
<td>180 days</td>
</tr>
<tr>
<td>Copper</td>
<td>Glass</td>
<td>8 oz</td>
<td>Grab</td>
<td>Ice (4°C)</td>
<td>180 days</td>
</tr>
<tr>
<td>Lead</td>
<td>Glass</td>
<td>8 oz</td>
<td>Grab</td>
<td>Ice (4°C)</td>
<td>180 days</td>
</tr>
<tr>
<td>Mercury</td>
<td>Glass</td>
<td>8 oz</td>
<td>Grab</td>
<td>Ice (4°C)</td>
<td>180 days</td>
</tr>
<tr>
<td>Nickel</td>
<td>Glass</td>
<td>8 oz</td>
<td>Grab</td>
<td>Ice (4°C)</td>
<td>180 days</td>
</tr>
<tr>
<td>Zinc</td>
<td>Glass</td>
<td>8 oz</td>
<td>Grab</td>
<td>Ice (4°C)</td>
<td>180 days</td>
</tr>
</tbody>
</table>

1. Samples initially stored in a cooler at 4°C.

Impoundment Bathymetric Survey and Sediment Profile
For the bathymetric mapping, sediment surface depths will be measured by dropping a secchi disc until it rests on top of the sediment and recording the water depth. The measurement will then be transferred to a steel rod, indicated by sliding a zip-tie to the appropriate distance from the bottom of the rod. For the sediment profile, the steel rod will then be driven into the sediment until the firm bottom is reached. The second zip-tie will be slid to the water level, thereby
indicating the depth of sediment with the distance between zip-ties. Measurements will be recorded in a field book with location identifiers, a base map noting the location of data collection and the associated depths for each.

### 2.3 Sample Handling and Custody Requirements

Detailed sampling and handling procedures are outlined in the Standard Operating Procedures provided in Appendix D. The five steps to sample handling and custody are described below.

**Completion of Field Data Sheets**

Field data sheets will be completed at the sample site during sampling activities and will include the following general information: site name and location, names of field team personnel, sample date/time collection, weather conditions, and comments. Upon filling each jar for laboratory analysis, the team member will place a check mark in the appropriate box for sieve analysis, total organic carbon, extractable petroleum hydrocarbons, and heavy metals to keep track of laboratory samples. The field team will also record the location and approximate depth of the samples taken. A sample field data sheet is provided in Appendix E.

**Sample Labeling**

Sample labels will be placed on clean, dry jars in advance of sampling. The laboratory will provide sample labels. An example label is provided in Appendix E. Each sample label will contain the following information once sampling is complete: sample name, location, sampler’s initials, date and time, preservation, and lab analysis to be performed.

**Sample Transport and Chain of Custody**

The samples will be placed in coolers on ice and transported to Geolabs subsequent to sample collection and within the sample holding times by laboratory courier. Once sampling is completed, the Field Team Leader will compile data sheets and samples that will be submitted for laboratory analysis. The containers will be inspected to ensure they are sealed and labeled appropriately. A chain of custody will be used to track the samples from the collection point through analysis and data management. The Field Team Leader will sign Chain of Custody records before samples are relinquished to the laboratory courier. The receiving laboratory will also sign the records. The Field Team Leader, Monitoring Coordinator and the laboratory analyzing the sample will maintain copies of the Chain of Custody. Appendix E contains the sample Chain of Custody form, which includes the following information: general project information, sample identification, date of collection, time of collection, sample matrix, sample type, sample analysis, and additional comments as appropriate.

**Sample Disposal**

The samples analyzed in the laboratory will be disposed in accordance with Massachusetts Law and the Standard Operating Procedures (SOP) of Geolabs.

**Sample Storage**

No samples will be stored for later use in this project.
2.4 Analytical Methods Requirements

Field measurements and data collection will be performed in accordance with the SOPs provided in Appendix D. The operating manual for the In-Situ Troll 9500 is also provided in Appendix D. Table 2.4-1 provides a summary of field measurements indicators and methods.

<table>
<thead>
<tr>
<th>Indicator</th>
<th>Responsible Organization</th>
<th>Method</th>
<th>Reporting Units</th>
<th>Modifications or options</th>
</tr>
</thead>
<tbody>
<tr>
<td>Turbidity</td>
<td>CEI / BRPC</td>
<td>In-Situ Troll 9500</td>
<td>NTU</td>
<td>None</td>
</tr>
<tr>
<td>Depth to Bottom</td>
<td>CEI / BRPC</td>
<td>Secchi Disk</td>
<td>ft</td>
<td>None</td>
</tr>
<tr>
<td>Depth of Sediment</td>
<td>CEI / BRPC</td>
<td>Rod and zip-ties</td>
<td>ft</td>
<td>None</td>
</tr>
</tbody>
</table>

Laboratory analyses will be performed in accordance with Geolabs QA/QC procedures, provided in Appendix F. Table 2.4-2 provides a summary of laboratory analytical indicators and methods.

<table>
<thead>
<tr>
<th>Indicator</th>
<th>Responsible Organization</th>
<th>Method</th>
<th>Reporting Units</th>
<th>Modifications or options</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sieve Analysis with Hydrometer</td>
<td>Geolabs</td>
<td>ASTM C136 / ASTM C117</td>
<td>gradation by %</td>
<td>None</td>
</tr>
<tr>
<td>Total Organic Carbon</td>
<td>Geolabs</td>
<td>SW-846 9060</td>
<td>mg/kg</td>
<td>None</td>
</tr>
<tr>
<td>Extractable Petroleum Hydrocarbons</td>
<td>Geolabs</td>
<td>MADEP EPH</td>
<td>ug/kg</td>
<td>None</td>
</tr>
<tr>
<td>Arsenic</td>
<td>Geolabs</td>
<td>SW-846 6010B</td>
<td>mg/kg</td>
<td>None</td>
</tr>
<tr>
<td>Cadmium</td>
<td>Geolabs</td>
<td>SW-846 6010B</td>
<td>mg/kg</td>
<td>None</td>
</tr>
<tr>
<td>Chromium</td>
<td>Geolabs</td>
<td>SW-846 6010B</td>
<td>mg/kg</td>
<td>None</td>
</tr>
<tr>
<td>Copper</td>
<td>Geolabs</td>
<td>SW-846 6010B</td>
<td>mg/kg</td>
<td>None</td>
</tr>
<tr>
<td>Lead</td>
<td>Geolabs</td>
<td>SW-846 6010B</td>
<td>mg/kg</td>
<td>None</td>
</tr>
<tr>
<td>Mercury</td>
<td>Geolabs</td>
<td>SW-846 7471A</td>
<td>mg/kg</td>
<td>None</td>
</tr>
<tr>
<td>Nickel</td>
<td>Geolabs</td>
<td>SW-846 6010B</td>
<td>mg/kg</td>
<td>None</td>
</tr>
<tr>
<td>Zinc</td>
<td>Geolabs</td>
<td>SW-846 6010B</td>
<td>mg/kg</td>
<td>None</td>
</tr>
</tbody>
</table>
2.5 Quality Control Requirements

Table 2.5 contains field and laboratory QC tests and samples for each indicator. For total organic carbon analysis, the laboratory analyzes a laboratory control spike where acceptable recovery limits are between 80% and 120%. For sieve analysis, the laboratory will perform a duplicate in which the relative percent difference must be less than or equal to 20%. QA/QC frequencies for extractable petroleum hydrocarbons and metals analyses are provided in Appendix F as part of laboratory quality control protocols and SOPs.

Table 2.5. Quality Control Checks¹

<table>
<thead>
<tr>
<th>Indicator</th>
<th>Field Quality Control Checks</th>
<th>EPA Method</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sieve Analysis with Hydrometer</td>
<td>Sampler is clean, sample is transferred immediately from sampler to sample container, container is properly sealed.</td>
<td>SW-846 Method 9060</td>
</tr>
<tr>
<td>Total Organic Carbon</td>
<td>Sampler is clean, sample is transferred immediately from sampler to sample container, container is properly sealed and immediately placed on ice.</td>
<td>SW-846 Method 9060</td>
</tr>
<tr>
<td>Extractable Petroleum Hydrocarbons</td>
<td>Sampler is clean, sample is transferred immediately from sampler to sample container, container is properly sealed and immediately placed on ice.</td>
<td>SW-846 Method 8100</td>
</tr>
<tr>
<td>Metals (not including mercury)</td>
<td>Sampler is clean, sample is transferred immediately from sampler to sample container, container is properly sealed and immediately placed on ice.</td>
<td>SW-846 Method 6010B</td>
</tr>
<tr>
<td>Mercury</td>
<td>Sampler is clean, sample is transferred immediately from sampler to sample container, container is properly sealed and immediately placed on ice.</td>
<td>SW-846 Method 7471A</td>
</tr>
</tbody>
</table>

¹ The QA/QC Officer and Field Team Leader will evaluate the field and lab data when available to determine if the QC samples reveal sampling or analytical problems.
### 2.6 Equipment Testing, Inspection and Maintenance Requirements

#### Table 2.6-1. Field Equipment Inspection and Maintenance

<table>
<thead>
<tr>
<th>Equipment Type</th>
<th>Inspection Frequency</th>
<th>Type of Inspection</th>
<th>Available Parts</th>
<th>Maintenance Frequency</th>
</tr>
</thead>
<tbody>
<tr>
<td>In-Situ Troll 9500</td>
<td>See installation SOP in Appendix D.</td>
<td>Working order, integrity, cleanliness, and properly calibrated according to manufacturer specifications.</td>
<td>Spare batteries. Parts available through supplier.</td>
<td>Annually or as needed.</td>
</tr>
<tr>
<td>Probe Sampler</td>
<td>Prior to each sampling event</td>
<td>Instrument is clean.</td>
<td>Spare sampler.</td>
<td>Annually or as needed.</td>
</tr>
<tr>
<td>Secchi Disk</td>
<td>Prior to each sampling event</td>
<td>Working order, integrity, cleanliness.</td>
<td>Spare secchi disk.</td>
<td>Annually or as needed.</td>
</tr>
<tr>
<td>Steel rod and zip ties</td>
<td>Prior to each sampling event</td>
<td>Instrument is clean.</td>
<td>Spare zip ties.</td>
<td>Annually or as needed.</td>
</tr>
<tr>
<td>Cooler</td>
<td>Prior to each sampling event</td>
<td>Cooler is clean; ice packs.</td>
<td>None necessary.</td>
<td>Annually or as needed.</td>
</tr>
</tbody>
</table>

#### Table 2.6-2. Laboratory Equipment Inspection and Maintenance

<table>
<thead>
<tr>
<th>Equipment Type</th>
<th>Inspection Frequency</th>
<th>Type of Inspection</th>
<th>Available Parts</th>
<th>Maintenance Frequency &amp; Recordkeeping</th>
</tr>
</thead>
<tbody>
<tr>
<td>Lab Benches</td>
<td>Each day of use</td>
<td>Cleanliness, chips, cracks.</td>
<td>Patch or filler kit</td>
<td>Clean and disinfect before each analysis date.</td>
</tr>
<tr>
<td>Drying Oven</td>
<td>Each day of use</td>
<td>Proper operation, cleanliness.</td>
<td>Per manufacturer</td>
<td>Semi-annually or as needed. Logbook notation.</td>
</tr>
<tr>
<td>Analytical Balance</td>
<td>Each day of use</td>
<td>Calibration verified at range of use.</td>
<td>Per manufacturer</td>
<td>Semi-annually or as needed. Logbook notation.</td>
</tr>
<tr>
<td>Inductively Coupled Plasma – Atomic Emissions Spectrometry Instrument</td>
<td>Each day of use</td>
<td>Calibration verified daily or more often as needed.</td>
<td>Per manufacturer</td>
<td>Semi-annually or as needed. Logbook notation, instrument output as paper files.</td>
</tr>
</tbody>
</table>
2.7 Equipment Calibration and Frequency Requirements

Table 2.7 outlines laboratory and field calibration procedures. Field calibration procedures are included in the Turbidity Monitoring SOP in Appendix C. The laboratory QA/QC manual is included in Appendix F of this QAPP.

**Table 2.7. Field and Laboratory Calibration Procedures**

<table>
<thead>
<tr>
<th>Equipment Type</th>
<th>Inspection and Calibration Frequency</th>
<th>Standard of Calibration Instrument Used</th>
<th>Logbook Notation</th>
<th>Corrective Action</th>
<th>Responsible Party</th>
</tr>
</thead>
<tbody>
<tr>
<td>Turbidimeter</td>
<td>Weekly for weeks 1 – 4 of monitoring, monthly or as needed thereafter</td>
<td>Manufacturers’ standards (instrument calibrates to standard solution via manufacturer’s software)</td>
<td>Yes</td>
<td>Calibrate</td>
<td>CEI/BRPC</td>
</tr>
<tr>
<td>Analytical Balance</td>
<td>Prior to use</td>
<td>Manufacturers’ standards</td>
<td>Yes</td>
<td>Replace/calibrate</td>
<td>Geolabs</td>
</tr>
<tr>
<td>ICP-AES Instrument</td>
<td>Daily calibration, instrument performance checks (IPC) after every 10 samples</td>
<td>±10% of calibration standard</td>
<td>Yes</td>
<td>Calibrate</td>
<td>Geolabs</td>
</tr>
</tbody>
</table>
## 2.8 Inspection / Acceptance Requirements of Supplies and Consumables

Table 2.8 outlines field supplies and recommended inspections. Field SOPs and procedures for calibrating the In-Situ Troll 9500 are contained in Appendix D.

<table>
<thead>
<tr>
<th>Supplies</th>
<th>Inspection Frequency</th>
<th>Type of Inspection</th>
<th>Available Parts</th>
<th>Maintenance</th>
<th>Responsible Party</th>
</tr>
</thead>
<tbody>
<tr>
<td>Field and lab sheets</td>
<td>Prior to each sampling event</td>
<td>Visual</td>
<td>Additional copies.</td>
<td>None necessary.</td>
<td>CEI / BRPC / Geolabs</td>
</tr>
<tr>
<td>Sample jars</td>
<td>Prior to each sampling event</td>
<td>Integrity, cleanliness.</td>
<td>One set of spare bottles.</td>
<td>None necessary.</td>
<td>CEI / BRPC / Geolabs</td>
</tr>
<tr>
<td>In-Situ Troll 9500</td>
<td>See installation SOP in Appendix D</td>
<td>Working order, integrity, cleanliness, and properly calibrated according to manufacturer specifications.</td>
<td>Spare batteries.</td>
<td>Annually or as needed based on inspections.</td>
<td>CEI / BRPC</td>
</tr>
<tr>
<td>Probe sediment sampler with slide hammer</td>
<td>Prior to each sampling event</td>
<td>Instrument and threaded connections are clean.</td>
<td>Spare plastic sleeves for sample collection.</td>
<td>Clean prior to each sampling event.</td>
<td>CEI / BRPC</td>
</tr>
<tr>
<td>Secchi Disk</td>
<td>Prior to each sampling event</td>
<td>Instrument is clean; mechanism is working correctly.</td>
<td>Spare disk.</td>
<td>Clean prior to each sampling event.</td>
<td>CEI / BRPC</td>
</tr>
<tr>
<td>Rod and zip-ties</td>
<td>Prior to each sampling event</td>
<td>Instrument is clean.</td>
<td>None necessary.</td>
<td>Clean prior to each sampling event.</td>
<td>CEI / BRPC</td>
</tr>
<tr>
<td>Cooler</td>
<td>Prior to each sampling event</td>
<td>Cooler is clean; ice packs.</td>
<td>None necessary.</td>
<td>Clean prior to each sampling event.</td>
<td>CEI / BRPC / Geolabs</td>
</tr>
</tbody>
</table>
2.9 Data Acquisition Requirements

For field sampling and data collection efforts, maps will be prepared using MassGIS data layers to help to identify sampling or data collection locations and the general land features in the area. Additional data will be obtained through the New England Interstate Water Pollution Control Commission (NEIWPCC) for the watershed model, AVGWLF. The data obtained from NEIWPCC was compiled from various data layers available through MassGIS and adapted for the input format required by AVGWLF.

The above-referenced sources provide state or federal databases with accurate information suitable for this study. Data collected during this project will be used to determine potential improvements in water quality through best management practices.

Additional field reconnaissance efforts will be performed by CEI and BRPC staff familiar with the area and experienced in conducting a watershed assessment. Watershed data such as land use types, additional sources of sediment inputs, and sites with a higher erosion or sedimentation potential will be compared to existing MassGIS data, as a check for watershed modeling accuracy. Adjustments will be made to the watershed model inputs in cases where significant disparities are observed between the acquired GIS data and the watershed survey. These adjustments will be tabulated to record each case where observed conditions deviate from spatial input data, the change made and the basis for the change.
2.10 Data Management

The field and laboratory data collected during this project will be managed according to the following stages:

**Raw Data**
Field and lab data sheets will be signed and checked for completeness by the Field Team Leader and Laboratory QA Officer. Omissions or apparent errors will be verified with the Field Team. Chain of Custody forms will follow the samples and the Laboratory QA Officer will inspect data sheets as soon as they are received. Sample receipt and/or analytical anomalies will be identified in the Narrative Section of the final summary report. Final summary reports are reviewed by Laboratory QA Officer or approved signatory prior to final submission. If a problem is suspected at this time, the Field Team Leader and/or samplers will be contacted. Lab analysts will flag data and comment on samples that did not arrive cool or have questionable characteristics. Data sheets and Chain of Custody forms will be sent to the Project Manager, QA/QC Officer, Monitoring Coordinator, and Field Team Leader as soon as the results are in.

**Data Entry and Validation**
The field and laboratory data will be entered into a spreadsheet (MS Excel). The Field Team Leader / Monitoring Coordinator will check the tabulated data against the field and lab sheets to assure that data has been entered correctly. The data will be critically reviewed by the Project Manager and QA/QC Officer for reasonableness, correspondence with data quality objectives, and appropriate qualification or censoring of suspect data.

**Data Storage**
Original field data sheets will be maintained with CEI. Laboratory data sheets and Chain of Custody forms will be maintained with Geolabs. Copies of original documents will be provided to MA DEP and BRPC for inclusion in the final report. Electronic files associated with the final report including sampling results will be stored with CEI for distribution as appropriate. Electronic files are stored on secure servers that are backed up several times each week. Spatial field data downloaded from the GPS and secondary spatial data used for the AVGWLF model or for mapping purposes will be maintained by CEI on a secure server that is backed up once per week. Data downloaded from the turbidimeter will be copied to backup CDs by field staff according to the provided SOP in Appendix D. A copy of each data download CD will be kept with associated project files by both BRPC and CEI.
3.0 Assessment and Oversight

3.1 Assessments and Response Actions
The project team member serving as Field Team Leader, Monitoring Coordinator and QA/QC Officer will be responsible for the following assessments and response actions:

- Observe the sampling team to assess their understanding of the written procedures both after the training session and after each sampling round. If deviations from the procedures are observed, they will be corrected and an assessment made as to whether previously collected data is suspect.
- Oversee data collection activities and handle corrective actions that pertain to data collection and data entry.
- Inform the Project Manager with the results of corrective actions.
- Review the field data sheets and tabulated laboratory data for information that pertains to data quality objectives throughout the project, particularly immediately after each sampling round and when laboratory data results are available.
- Review the watershed reconnaissance observations with the Technical Advisor to determine whether corrective action pertaining to spatial land use data is necessary.

The Project Manager will be responsible for oversight and follow-up on corrective actions reported by the Field Team Leader, Monitoring Coordinator, and QA/QC Officer throughout the project.

The Laboratory QA/QC Officer will provide the laboratory assessment procedures after each sampling round and during data analysis. The Laboratory QA/QC Officer will discuss sampling issues with the Field Team Leader / Monitoring Coordinator as necessary.

The Technical Advisors will assist with these tasks throughout the project as appropriate.
3.2 Reports

A summary memo will be prepared after the completion of each sampling and significant field data collection event, once data results are received or generated but at a minimum at least prior to further sampling or field data collection event. The memo, prepared by the Project Manager and project team member serving as Field Team Leader, Monitoring Coordinator and QA/QC Officer, will summarize the sampling/monitoring event and note problems that occurred with recommendations for improvement prior to the next sampling event.

Monthly and/or quarterly reports will be distributed to the City of Pittsfield, BRPC, MA DEP and EPA for comment and information purposes as part of the 604(b) reporting requirements. As appropriate, the project quality assurance information will be included.

Final sampling/monitoring results will be incorporated into the final project report required by the MA DEP 604(b) Grant Program at the completion of the project. This report will contain the following:

- Executive Summary
- Project Description
- Existing Data Assessment
- Environmental Monitoring
- QA/QC Issues (field and laboratory) and QAPP Deviations
- Evaluation of Data and Modeling
- Recommendations and Conclusions
- Data Gaps
- References and Further Reading

CEI will be responsible for interpreting the laboratory results, compiling monitoring data sheets, and providing the technical information to be included in the report. BRPC will be responsible completing the final report encompassing the above sections. Draft copies will be distributed to the City of Pittsfield, CEI, MA DEP and EPA for review and comment prior to finalization and approval.
4.0 Data Validation, Usability and Model Application

4.1 Data Review, Validation and Verification Requirements

Data will be reviewed by the Project Manager, the Field Team Leader and Monitoring Coordinator, and QA/QC Officer, and the MA DEP Project Officer to determine if they meet the QAPP objectives. The Field Team Leader will initially review field data for completeness as soon as possible after collection. If data review indicates a problem with a particular sample, it is possible that another sample round will be collected at the site. The Project Manager and project team member serving as Monitoring Coordinator, Field Team Leader and QA/QC Officer will determine the appropriateness and need for such an action.

The Project Manager and QA/QC Officer will review laboratory data for completeness as soon as possible after analysis. Data on quality control measures should accompany the sample data. If discrepancies that exceed the data quality objectives are observed, re-analysis may be possible, as long as additional sample material is available and holding times have not been exceeded.

The Project Manager and QA/QC Officer using available QC data will review the combined data set after the field collection data. Deviations will be flagged, incomplete data will be noted, and sampling staff will be reminded that complete data logs are necessary. Calculations will be spot-checked. QC results that deviate from the data quality objectives will call the validity of the individual data or related data into question. The final decision on whether to include or reject the data will be made by the Project Manager and QA/QC Officer and the MA DEP Project Officer.

Data collected for the AVGWLF model will be validated using information collected via watershed reconnaissance. The level of quality in the available model data from NEIWPCC is appropriate for the model’s intended role within this project – to determine a gross estimate of sediment load at each of the two impoundments.
4.2 Validation and Verification Methods

Each verifying group will examine data for logical consistency, as presented in Table 4.2. If inconsistencies are found, an attempt will be made to determine whether the data is in error. For example, field notes taken at the time of the sampling event might indicate possible reasons for inconsistency. Apparent problems will be noted in the final reports as appropriate.

Table 4.2. Validation and Verification Procedures

<table>
<thead>
<tr>
<th>Verifying Group</th>
<th>When</th>
<th>Activity</th>
<th>Possible Corrective Measures and Notification</th>
</tr>
</thead>
<tbody>
<tr>
<td>Field Team Leader</td>
<td>Sampling or field data collection day – when field staff turns in their data sheets, chains of custody.</td>
<td>Collect, review field staff field sheets for outliers, illegible data entries, missing data, etc.</td>
<td>Discuss with field staff. Flag problems that are not correctable. Fill in known data.</td>
</tr>
<tr>
<td>Laboratory Project Manager</td>
<td>Sampling day – when samples are submitted, chains of custody, laboratory report.</td>
<td>Same as above for chain of custody. Check for samples that exceeded holding time, arrived in improper condition (i.e., too warm, improperly sealed).</td>
<td>Analytical issues will be identified and addressed by the Laboratory Area Supervisors. Sample receipt and/or analytical anomalies will be identified in the Narrative Section of the final summary report.</td>
</tr>
<tr>
<td>Monitoring Coordinator</td>
<td>As soon as possible after each sampling or field data collection date.</td>
<td>Review data/ observation, field sheets, and chain of custody sheets.</td>
<td>Discuss outliers or errors with other team members and correct those found.</td>
</tr>
<tr>
<td>Project Manager / QA/QC Officer</td>
<td>Shortly after each sampling or field data collection date. When data compilation has been completed. When QC data are reported.</td>
<td>Compare # of QC tests performed vs. # outlined in QAPP. Compare QC test results with targets or expected values. Spot-check calculations. Check for transcription errors. Check again for outliers.</td>
<td>Check to see if QC results sheets are missing. Discuss outliers or errors with other team members and correct those found. Re-run calculations. Re-run QC tests, if possible. Flag problems that are not correctable.</td>
</tr>
</tbody>
</table>
4.3 Reconciliation with Data Quality Objectives

The Project Manager and QA/QC Officer will compare the data with the objectives for precision, completeness, and accuracy provided in Section 1.7. The following data review decision tree (Figure 4.3-1) from the Massachusetts Volunteer Monitor’s Guidebook to Quality Assurance Project Plans (October 1, 2001) will serve as the basis for evaluating the quality of project data. Necessary corrective actions will be implemented, documented and initiated by the Project Manager and QA/QC Officer. Data quality objective failures due to equipment issues will result in the reassessment of calibration and maintenance procedures, with affected data so noted.

Figure 4.3-1. Data Review Decision Tree

* This step includes evaluating data in its usefulness for answering the study questions listed in Section 1.6 (four numbered questions) and Section 1.7 (three bulleted questions).
Appendix A

Project Personnel Sign-Off Sheet
Training Record
<table>
<thead>
<tr>
<th>Name</th>
<th>Sampling or Field Data Collection Performed</th>
<th>Date</th>
</tr>
</thead>
<tbody>
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<tr>
<td>Name</td>
<td>Topic or Activity(ies)</td>
<td>Instructor</td>
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</table>

FIELD TEAM TRAINING RECORD
QUALITY ASSURANCE PROJECT PLAN
WINDSOR & CADY BROOK SEDIMENT & TURBIDITY MANAGEMENT
FOR CLEVELAND RESERVOIR
Appendix B

Windsor & Cady Brooks
Impoundments and Diversion Structures
(Photos)
A Series Photos (4/21/09)

Windsor Brook Impoundment

Windsor Brook Intake Structure
A Series Photos (4/21/09)

Sediment at Windsor Brook Impoundment

Sediment at Windsor Brook Impoundment
View Downstream of Windsor Brook Impoundment

Combined Aqueduct to Cleveland Brook Reservoir
A Series Photos (4/21/09)

Cady Brook Impoundment

Cady Brook Intake Structure
A Series Photos (4/21/09)

Sediment at Cady Brook Impoundment

Sediment at Cady Brook Impoundment
A Series Photos (4/21/09)

View Downstream of Cady Brook Impoundment
<table>
<thead>
<tr>
<th>MONTH</th>
<th>TASK</th>
</tr>
</thead>
<tbody>
<tr>
<td>JAN</td>
<td>Task #1. Quality Assurance Project Plan (QAPP)</td>
</tr>
<tr>
<td>FEB</td>
<td>Task #2. Characterization of Turbidity in Windsor &amp; Cady Brook Diversion Impoundments</td>
</tr>
<tr>
<td>MAR</td>
<td>Task #3. Evaluation of Potential Sediment Sources</td>
</tr>
<tr>
<td>APR</td>
<td>Task #4. Estimating Watershed Sediment Volumes</td>
</tr>
<tr>
<td>MAY</td>
<td>Task #5. Evaluation of BMPs for Managing Sediment &amp; Turbidity</td>
</tr>
<tr>
<td>JUN</td>
<td>Task #6. Reporting</td>
</tr>
</tbody>
</table>
Appendix D

Standard Operating Procedures:

Bathymetry and Sediment Thickness
Sediment Sampling
Sediment Yield Model
Turbidity Monitoring
Operating Manual for In-Situ Troll 9500
SOP Bathymetry and Sediment Thickness

Equipment:
- Canoe or comparable small water craft (coordinate with BRPC staff and Pittsfield DPW for deployment)
- US Coast Guard approved life jackets – required for all personnel using watercraft
- Rope (minimum length 200 feet), for use in securing watercraft to shore, if necessary to assist in positioning craft for survey
- 4-foot wooden stakes for use as range finders on shore
- Survey level (Topcon Green Label AT-G4 Auto Level, or comparable equipment)
- Survey tripod
- Stadia rod (English decimal units)
- 200-ft survey tape (English decimal units)
- Secchi disk and cable
- Nylon measuring tape marked in feet and tenth-foot intervals at least 10 feet in length
- At least two spring-clip clothes pins
- Steel rod with two zip ties for probing to measure depth of sediment. Provide sufficient rod length to penetrate approximately 10 feet.
- GPS receiver/datalogger (Trimble GeoExplorer CE, or comparable equipment)
- Laptop computer
- CDs for recording backup to laptop data files
- Backup batteries for laptop and GPS equipment
- Field book and pencils
- Copy of QAPP and this SOP

Prior to mapping event:
- Confirm that Bench Mark and staff gages have been installed as specified under other protocol(s).
- Coordinate with DPW personnel for deployment of water craft, paddles/oars and safety vests at the site prior to arrival of field crew
- Coordinate with BRPC for field crew staff
- Prepare equipment required for the visit
- Verify weather predictions for date of field work, to confirm continued base flow conditions at the time of the work. Work shall be initiated following minimum dry weather flow of three days, unless local DPW otherwise confirms that the overflow spillway is operating under base flow conditions. Reschedule field work if storm conditions occur or are predicted within three days preceding the event and on the day of the event.
Field procedure:

- Review applicable provisions of this SOP and the QAPP with field crew at the site.
- Place range finding stakes at sufficient intervals to assist in positioning watercraft for survey measurements.
  - Place two stakes on the same side of the impoundment at each cross section, approximately perpendicular to the centerline of the impoundment.
  - Place each pair of stakes at approximately 50-foot intervals, readily observable from the water. The two stakes in each pair should be separated by a distance of about 10 feet or more, as permitted by field conditions, to facilitate aligning the craft for taking measurements.
- Launch water craft in a location where it can be safely entered by all personnel.
  - At least two persons are required to operate the craft and obtain data measurements.
  - All persons in craft must wear life vests.
  - If flow conditions affect craft maneuverability, use rope(s) to secure craft to large tree(s) or other fixed objects on shore, to assist in positioning and maintaining position of craft to conduct measurements.
  - Deploy rod and secchi disk for sediment probes, sediment sampler (as applicable), GPS equipment, and field books and related supplies in the water craft.
- Initiate mapping by reading the water level on the staff gage (previously installed under other protocol) at the beginning of the survey. All depth probes and sediment probes will be read from the elevation of the water surface.
- Periodically read the staff gage during the survey event, and record the time and the elevation in the field book (alternatively, the GPS data logger may be used for this notation). Recommended intervals between readings should be about one hour.
- Align the water craft with the pair of range stakes representing the first cross section.
  - Start at one side of the impoundment and maneuver across the impoundment in line with the range stakes.
  - Take readings at approximately 10 to 15 foot intervals. Record time and position using the GPS positioning feature, and log depth to top of sediment using secchi disk, and approximate depth of soft sediment using steel rod.
  - Drop secchi disk into water. When the disk rests at the sediment surface, hold the line taut and attach a clothes pin on the line at water level.
o Withdraw secchi disk and use nylon measuring tape to read depth along the cable. Record depth in field book or GPS data logger.

o Use tape measure to set one of the zip-ties on the steel rod to the depth just recorded using the secchi disk.

o Lower rod into the water with the remaining zip-tie in a location that will remain above water level. Force rod as far as possible until firm bottom is encountered, or to maximum depth of the equipment, or to the maximum depth safe under conditions encountered. Slide the remaining zip-tie to the water level.

o Withdraw the rod and measure the distance between the two zip-ties as the measured depth of sediment.

o Measurements will be taken from the secchi disk and probe to the nearest tenth foot.

o Use the field book to note any conditions that prevent a complete measurement.

• Repeat this procedure at each cross section.

• At the completion of this procedure, take a final reading of the time and the water elevation on the staff gage.

• Where sediment deposition is observed to exceed the water elevation at the time of survey, obtain elevations on the top of sediment using the level and stadia rod. If at all possible, access this area from the shore, rather than from the watercraft. Exercise care in traversing soft sediment. If the surface is inaccessible, use the GPS equipment to locate the sediment deposit “shoreline” (intersection of the sediment with the water-line), and estimate the height of sediment above this level. All level/stadia rod readings shall be referenced to the bench mark established at the site under other protocols.

Data reduction:

• GPS data shall be downloaded at the end of the day to the laptop. A backup CD shall be made immediately following download. BRPC personnel shall take custody of the CD. CEI personnel will reduce digital data from the laptop file.

• Upon return to the office, CEI personnel will download GPS position, depth, and sediment thickness data to an AutoCAD file, to create descriptive drawing layers indicating the measurement points, bottom surface bathymetry, and recorded sediment thickness.

• Measurements relative to water surface will be mathematically converted to bench mark datum.

• The drawing layer depicting these features will be registered with a base drawing of the impoundment. The base drawing will be developed from existing record
drawings of the diversion structure impoundments supplied by the City of Pittsfield.

- Sediment profiles will be generated at the cross sections from the reduced data and drawing information.
SCHEMATIC PLAN
SCALE 1"=30'

SUPPLEMENTAL DEPTH MEASUREMENTS NEAR SPILLWAY, IF FIELD CONDITIONS ALLOW (LOCATIONS MAY VARY)

LEGEND

C 54" DIA. CONDUIT

12" RIP-RAP
SCHEMATIC PLAN

SCALE 1"=30'
SOP Sediment Sampling

Equipment:
- Containers for conveying sediment samples to laboratory
- Small containers or Zip-lock bags for collecting representative portions of the samples for visual analysis in the office.
- Shovel
- AMS Equipment Soil Core Sampler (or comparable alternative), with slide hammer and expendable plastic liners
- Clean stainless steel bucket(s) for field compositing samples
- Soap for decontamination (Alconox or equivalent)
- Deionized water
- GPS receiver/data logger
- Field note book and pencils/pens
- Field sampling forms, chain of custody forms, labels
- This sampling will be conducted concurrently with bathymetric mapping – see other protocol for appropriate equipment.
- Copy of the QAPP and this SOP

Prior to site visit:
- Coordinate with BRPC for field crew staffing, and to integrate this activity with the bathymetric and sediment thickness mapping
- Contact laboratory to confirm sample size required for specified analytes, and verify procedures and schedule for collection and delivery of the samples.
- Obtain and prepare equipment for the field sampling.
- Participating personnel will review the QAPP and this SOP

Sample Collection Procedure:
- Upon arrival at the site, review SOP for sediment sampling with the field team, and coordinate this activity with the bathymetric and sediment mapping.
- Up to two sediment samples will be collected at each impoundment. During project scope development, CEI observed that each impoundment has accumulated sediment to and above the normal pool elevation. Sediment samples will be obtained from these sediment deposits.
  - Record each sample location, date, and conditions of sampling using the GPS data logger, with supplemental notations as warranted in the field notebook. Designate each sample with an alphanumeric sample name, to identify the impoundment and the location of the sample.
As a guide, the sediment sample from the mid channel deposit should be collected from the approximate center of the deposit, or toward the downstream end.

Refer to the soil core sampler user instructions for the assembly and use of the core sampler.

Remove the top 6 to 8 inches of material using the shovel, to expose the underlying sediment. From this underlying sediment, extract sufficient material to comprise the required sample size and place directly into the sample container.

At each sampling location, insert the sampling device into the exposed sediment, and advance the device into the material by physically pushing the rod. If necessary, use the slide hammer to assist in advancing the probe. When the probe has fully entered the sediment deposit, measure depth, withdraw the probe, remove the plastic liner.

In the field notebook, describe the sediment (color, texture, observed stratification) and note sample depth.

Deposit the collected material into a clean bucket.

Replace the plastic liner, if still serviceable, into the coring device, and advance the probe deeper into the same hole a sufficient distance to fill the sampler. Withdraw this portion of the sample, record a description of the sediment and the depth of sample, and place the material in the mixing bucket.

If the liner is not serviceable, replace with a new liner.

Repeat the procedure until the probe encounters refusal or until sufficient sediment has been retrieved to composite the required sample size, allowing for separating a sub-sample for visual observation in the office.

If refusal is encountered prior to acquiring a sufficient sample size, start a second core in the near vicinity of the first core, and repeat the procedure until sufficient material is obtained.

Thoroughly mix the sample portions in the bucket, and then transfer to the sampling containers:

- Primary container for delivery to the laboratory.
- Small companion sample in Zip-lock bag for qualitative, visual examination in the office.

Securely close and label each sample.

Store the samples immediately on ice or at 4°C.

Prior to collecting further samples, decontaminate the steel bucket and tip of the probe using Alconox and deionized water.

Use a fresh plastic liner (or set of liners) for each sample location.
Sample Analysis:

- Continue to store the samples on ice or at 4°C.
- Coordinate laboratory pickup of the sediment samples such that the laboratory is provided adequate time to analyze samples within the shortest holding time of all analyses (14 days).
- Fill out a chain of custody form with appropriate information. At time of courier pick-up, both the courier and relinquishing field team member should sign to confirm the date and time at which this occurred.
- Samples shall be analyzed for the following:
  - Sieve analysis with hydrometer
  - Total organic carbon
  - Extractable petroleum hydrocarbons
  - Heavy metals including arsenic, cadmium, chromium, copper, lead, mercury, nickel and zinc
- For analytical procedures, see Laboratory QA/QC procedures provided in Appendix E
SOP Sediment Yield Model

Software:
- CEI anticipates that the primary software for this work element will comprise the model “ArcView Generalized Watershed Loading Function” (AVGWLF), a Geographic Information Systems (GIS) interface developed by Penn State Institutes of Energy and the Environment for the GWLF model, or a comparable method of analysis. Additional information about this model can be found at the following web-site: [http://www.avgwlf.psu.edu/](http://www.avgwlf.psu.edu/)
- An associated model, PredICT (insert similar source information) that works in conjunction with this model may be used, if the study team deems it appropriate for assessing the effectiveness of potential management actions. However, this model component has limitations in the types and scale of BMPs that can be analyzed, and in the assignment of sediment removal rates to those BMPs. It is most useful for assessing application of certain agricultural management practices, which may not be appropriate management strategies for this particular watershed.
- As an alternative, selected alternative scenarios will either be modeled using AVGWLF, or the study team will employ simple computational estimates and spreadsheet analyses to evaluate BMPs (e.g., typical stormwater management BMPs) to assess sediment removal for individual devices, and these individual results can be aggregated for groups of BMPs to compare to baseline sediment estimates generated by AVGWLF.

Source Data:
- GIS data files have been incorporated into the Software format by NEIWPCC. The relevant files as listed below will be used in this study:
  - Streams
    Data type: line (stream segments)
    Source: National Hydrography Dataset
  - Weather Stations
    Data Type: point feature with associated Excel files containing daily temperature and precipitation data (1990 to 2004)
    Source: USGS topographic maps or aerial photos (for location), National Weather Service (for climate information)
  - Soils
    Data Type: polygon
    Source: U.S. General Soil Map, STATSGO data sets
- Physiographic Province (for rainfall erosivity coefficients)
  Data Type: polygon
  Source: Selker et al., 1990
- Land Use/Cover
  Data Type: grid
  Source: MassGIS Land Use Data (1999)
- Surface Elevation (Topography)
  Data Type: grid (Digital Elevation Model, 30-meter)
  Source: U.S. Geological Survey

The GIS data layers will be downloaded to develop base maps, with Land Uses and other pertinent features to be field confirmed through a “windshield” reconnaissance. Unless significant discrepancies are noted in the field, the default GIS data of the model will be used. Minor land use differences (such as individual houses) will not appreciably affect results, and will not altered in the base files.

Rainfall data files are included in the model Software NEIWPCC files. These files will be used to generate rainfall and stream flow data for the study watersheds. As a default, if none of the weather stations included in the provided data set lies within the watershed, the model selects the two closest weather stations from the available set and calculates mean values as model inputs. For the Windsor and Cady Brook watersheds, AVGWLF will by default use weather data collected at National Weather Stations in Amherst, MA and Grafton, NY.

**Modeling Runs:**

- The model will be run for the current condition using the default GIS (as amended based on field reconnaissance) and rainfall base files.
- The model will be run for hypothetical “virgin forest” conditions, to assess the impact of human activity on the watershed, and provide an indication of the sensitivity of the watershed to such activity.
- As noted above, the model may also be used to generate sediment estimates associated with proposed management scenarios, if in the judgment of the study team the model will provide meaningful results. Otherwise, the current condition and “virgin forest” modeling results will serve as a baseline with which to compare the sediment removal performance of various BMP scenarios, with their performance evaluated using other accepted engineering computational methods.
- The model is being used as a computational tool, to generate an order of magnitude estimate of sediment quantity for the study watersheds. The model uses the USDA Universal Soil Loss Equation, a widely accepted computation

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procedure for estimating soil erosion and sediment delivery. (The use of the model in this way is analogous to using TR-55 and TR-20 for estimating peak flows and runoff volumes for stormwater management planning.)

- The model will not be calibrated to known sediment quantities. The detailed study of baseline sediment concentrations and loads necessary for such a calibration is well outside the scope of this study.
- Modeling will generate average flow data and sediment quantities for each watershed on a monthly and annual basis.
SOP Turbidity Monitoring

Equipment

- Staff gage, four feet in length marked in foot, tenth-foot and hundredth-foot intervals
- Post (8-foot long steel post)
- 1” x 4” x 7’ pressure treated lumber board, for mounting staff gage
- PVC pipe, 4-inch diameter, 7 feet long
- PVC pipe cap to fit 4-inch diameter pipe
- Turbidimeter (In-Situ Troll 9500 or equivalent)
- Instrument control software (Win-Situ 4 or equivalent)
- Communication adapter cable (USB) for turbidimeter data downloads
- Calibration solution for turbidity
- Benchtop turbidimeter (Hach 2100 N) housed at water treatment plant
- Hand tools (e.g., drill) required for mounting equipment on diversion structure OR posts
- Communication/suspension cable (RuggedCable or equivalent)
- Bolts and brackets required for mounting PVC pipe to concrete structure and anchoring turbidimeter inside PVC pipe
- Survey level (Topcon Green Label AT-G4 Auto Level, or comparable equipment)
- Stadia rod (English decimal units)
- Survey tripod
- Laptop computer
- CDs for recording backup to laptop data files
- Backup batteries for turbidimeter and laptop
- Field note book and pencils/pens
- Copy of the QAPP, this SOP and turbidimeter operating manual

Prior to site visit:

- Coordinate with DPW personnel for mounting staff gage and monitoring instrument to structure and for the use of the benchtop turbidimeter at the water treatment plant
- Coordinate with BRPC for field crew staff
- Verify weather predictions for date of field work, to confirm continued base flow conditions at the time of the work. Work shall be initiated following minimum dry weather flow of three days, unless local DPW otherwise confirms that the overflow spillway is operating under base flow conditions. Reschedule field work of storm conditions occur or are predicted within three days preceding the event and on the day of the event.
- Prepare equipment required for the visit, as listed above.
- Distribute and install instrument control software on field laptop and computers that will be used to interpret data.
Turbidity Pre-Calibration Procedure:

- Visit the Cleveland Reservoir water treatment plant with the calibration solution to be used for the field turbidimeter.
- Confirm with treatment plant staff that the benchtop turbidimeter has been calibrated according to typical schedule.
- Analyze a vial of the calibration solution to be used for the field instrument, conforming to the process used at the treatment plant.
- Compare the result with the standard solution value (in NTU).
  - If the values are not equal and the difference is greater than 10% of the calibration standard value, recalibrate the benchtop instrument. If recalibration does not correct this error, apply a secure label to the bottle of calibration solution with the observed turbidity reading and date.
  - If the values are not equal and the difference is 10% or less than the calibration standard value, apply a secure label to the bottle of calibration solution with the observed turbidity reading and date.
  - If the values are equal, apply a secure label to the solution bottle noting the date and observed turbidity (or standard value).
- Repeat this process prior to calibrating the field instrument if the bottle label with the calibration value is missing or a new bottle of solution is obtained.

Staff Gage Installation Procedure:

- Review applicable provisions of this SOP and the QAPP with field crew at the site.
- Establish an assumed Bench Mark elevation value or locate a pre-set Bench Mark to reference relative elevations, all to the nearest hundredth (0.01 foot).
- Pending confirmation of installation method with City of Pittsfield, mount staff gage directly to diversion structure next to intake. This will be done by drilling and setting concrete anchors, with staff gage sections attached to anchors using compatible screws. If attachment to structure proves infeasible, then install freestanding post next to structure with approximately two feet penetrating into ground. Mount staff gage on pressure treated board, and attach the board to post by screws inserted into the back of the board, through pre-drilled holes in post. Each staff gage is considered a temporary structure for the duration of the project.
- Measurement markings on the mounted staff gage should extend approximately one foot below the overflow spillway elevation.
- Establish relation between staff gage measurements and Bench Mark elevation using traditional survey methods with the survey level, stadia rod and tripod.
- As a precautionary measure in the case of a problem with the staff gage, set a secondary, accessible Bench Mark reference on structure such that water level can be determined using a measuring tape from the concrete platform above the intake. This bench mark
should be a level surface on top of the structure, or an indelible mark on the side of the structure, located so as to permit a direct measurement by tape from the bench mark to the water surface.

**Turbidity Monitor Installation Procedure:**

- Lay PVC pipe on a dry, workable surface. Hold instrument probe near one end and thread communication cable through the pipe. Secure the probe inside the pipe using an appropriately sized anchor(s) such that 8 inches extends beyond the end of the pipe.
- At the opposite end of pipe, anchor the cable to the inner wall using an appropriately sized anchor(s) or secure using cable grip with a hook anchored to inner wall.
- Install a hook or tie for storing the coiled remaining length of cord inside pipe.
- Place PVC pipe against the side of the diversion structure farthest from the dam spillway, perpendicular to level concrete slab and as close as feasible to the diversion structure inlet opening (opening with bar rack).
- Anchor the PVC pipe to the concrete with a brackets sufficient to secure the pipe in a fixed position.
- Position the PVC pipe through the brackets such that the pipe end lies approximately two feet above the bottom elevation of the intake weir (based on remaining length of pipe above concrete slab).
  - At Windsor Brook, bottom of pipe should be 5 feet below concrete platform.
  - At Cady Brook, bottom of pipe should be 6 feet below concrete platform.
- If mounting on the structure proves infeasible, the pipe may be secured to a mounting board, attached to a post, in similar manner to the installation of the staff gage.
- Before leaving site, coil the instrument cable and place inside the pipe. Cover with cap.
- Calibrate the turbidimeter to log water level relative to the bench mark according to the steps outlined in the method below.
- Set the turbidimeter clock and program to begin recording data via the instrument’s control software.
- Lower slowly into the water.
- With the communication adapter cable connected to the laptop, use control software to log an initial set of readings while allowing the instrument to stabilize to water conditions for approximately one hour. Examine data for stabilization.
- Verify water levels with staff gage readings. Make adjustments to the instrument calibration if necessary.
- Set up logging schedule using instrument control software according to operator’s manual. The schedule must specify the abovementioned parameters and a logging interval of 30 minutes. Initiate data logging.
- Verify logging schedule set-up by downloading data after one hour. Compare data with real-time readings and grab sample reading. Do not erase logged data.
At the end of the monitoring period, remove the turbidimeter by carefully dismantling the instrument set-up.

**Turbidimeter Calibration:**
*Note: Steps based on method provided in Section 18 of the Operator’s Manual.*

- Rinse the front end of the turbidimeter probe with clean water. Shake well to remove the rinse water and dry all but the sensor optical windows with a clean tissue.
- Examine the bottle of turbidity calibration standard for the adjusted standard value. If this label is missing, repeat the Turbidity Pre-Calibration Procedure. If the bottle does not contain enough solution for calibration, obtain a new bottle of solution and repeat the Turbidity Pre-Calibration Procedure.
- Pour the turbidity calibration standard into a beaker and insert the sensor end of the instrument into the solution so that windows of turbidity sensor are at least a quarter inch deep in solution. Gently agitate instrument to dispel air bubbles.
- Connect the turbidimeter to a laptop and establish a connection via control software.
- Select instrument in navigation window and select Turbidity in the Parameters list.
- Select Calibrate to open the calibration wizard.
- Note the adjusted calibration standard value affixed to the bottle and enter this value (rather than the manufacturer’s labeled value) during the step by step calibration process.
- When the process is complete, open the calibration report and save the file to the laptop and two backup CDs.
- Rinse the front end of the turbidimeter probe again with clean water.
- Leftover turbidity calibration standard may be disposed of onsite.

**Data Downloads and Turbidimeter Maintenance Procedures:**
- Train field staff in field use of the turbidimeter according to instructions in operator’s manual.
- For any field site visit during which data will be downloaded from the instrument, data must be saved on a laptop computer hard drive. While on-site, also make two backup CDs containing the downloaded raw data, for the project files at both BRPC and CEI. Verify that the laptop and each CD contain the complete data set in the field, prior to erasing data from the instrument.
- Visit the site at the end of Weeks 1, 2 and 4 following turbidimeter installation to inspect the instrument and download data. During each visit, verify current depth data with the staff gage reading.
- With two alkaline batteries, the instrument will likely not require battery replacement during the monitoring period. However, monitor battery life indicator via laptop to determine if proactive battery replacement is necessary.
- Repeat the previous two steps and download data at the end of each month following Week 4.
• Continue monitoring program with monthly data downloads again at Weeks 8, 12 and 16 for a total monitoring period of four months.
• Visit the site following any significant storm event during the monitoring period to ensure that staff gage is secure and turbidimeter is recording properly (repeat field checks for water level and turbidity as in previous instruction).
• Replace batteries in the turbidimeter according to the frequency determined during Weeks 1 through 4 of monitoring. Field crews should have spare batteries on hand at each field visit, in case replacement is necessary. Should batteries require replacement, recalibrate the instrument prior to logging further data. In addition, the instrument should be recalibrated following any power-off.
• Perform any necessary maintenance according to instructions in the operator’s manual.
• Prior to leaving the site, verify that instrument and staff gage set-ups are secure.
• To process data, export turbidity and depth data from the instrument software into an Excel-format spreadsheet. Plot turbidity on an appropriate time scale for comparison with data furnished by the City of Pittsfield.
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GENERAL DESCRIPTION OF THE MP TROLL 9500

Your new Multi-Parameter TROLL 9500 water quality probe uses the latest sensor and electronics technology to provide a robust, durable, and user-friendly instrument.

The MP TROLL 9500 logs data from up to 7 water-level and water-quality sensors, as well as built-in temperature and barometric pressure sensors. Many custom options and versions are available, so your instrument may not look exactly like those we have chosen to illustrate in this manual.

The MP TROLL 9500 provides the convenience of a laboratory-quality measurement instrument for field use, providing true in-situ monitoring of water level and water quality.

HOW TO USE THIS MANUAL

This operator’s manual is designed as both a start-up guide and a permanent reference for the features, uses, and applications of the Multi-Parameter TROLL 9500.

Section 1: Introduction to this Operator’s Manual and to In-Situ Inc.; contacting us for warranty and repair issues
Section 2: Description of components and features of the Multi-Parameter TROLL 9500
Section 3: Getting Started: setting up the software, connecting for the first time, Quick-Calibrating the sensors, and a summary overview of the setup to start collecting data
Section 4: Control Software: Win-Situ® 4 and Pocket-Situ 4
Section 5: Profiling
Section 6: How to set up tests and start logging data; extracting data to your PC; viewing and graphing data
Section 7: Monitoring Pressure (Water Level): Info on the two basic types of pressure sensors; setting up the pressure channel to obtain the most accurate readings for your altitude and latitude; how to display data as you wish to see it
Section 8: Temperature
Section 9: Barometric Pressure
Section 10: An overview of monitoring water quality with the MP TROLL 9500: Sensor installation and calibration, software options, calibration reports
Section 11: pH
Section 12: Conductivity
Section 13: Dissolved Oxygen: Polarographic (Clark cell) RDO® Optical Dissolved Oxygen
Section 14: Oxidation-Reduction Potential (ORP)
Section 15: Ammonium
Section 16: Chloride
Section 17: Nitrate
Section 18: Turbidity
Section 19: SDI-12 Operation
Section 20: Low-Flow Monitoring
Section 21: Care and Maintenance advice
Section 22: Troubleshooting: Look here first if you have problems making connections or calibrating . . .

The manual includes a Glossary and a comprehensive Index
CONVENTIONS

Throughout this operator’s manual you will see the following symbols.

✅ The check mark highlights a tip about a convenient feature of the MP TROLL 9500

⚠️ The exclamation point calls your attention to a requirement or important action that should not be overlooked

Typical usage questions and answers that we hope will help you gain a better understanding of your new Multi-Parameter TROLL 9500 and simplify its setup and operation

UNPACKING AND INSPECTION

Your MP TROLL 9500 was carefully inspected before shipping. Check the instrument for any physical damage sustained during shipment. Notify In-Situ and file a claim with the carrier if there is any such damage; do not attempt to operate the instrument.

Please save packing materials for future storage and shipping of your MP TROLL 9500. The shipping boxes have been performance-tested and provide protection for the instrument and its accessories.

Accessories may be shipped separately and should also be inspected for physical damage and the fulfillment of your order.

SERIAL NUMBER

The serial number is located on the large label on the Multi-Parameter TROLL 9500 body, and also on a small label inside the battery compartment. The serial number is programmed into the instrument and displayed when the instrument is connected to a PC running Win-Situ 4 or Pocket-Situ 4. We recommend that owners keep a separate record of this number. Should your Multi-Parameter TROLL 9500 be lost or stolen, the serial number is often necessary for tracing and recovery, as well as any insurance claims. If necessary, In-Situ maintains complete records of original owner’s names and serial numbers.

Serial numbers of the water-quality sensors are stored in sensor memory and displayed in the software when sensors are installed.

TO OUR CUSTOMERS . . .

Thank you for your purchase of an In-Situ product. We are glad you chose us and our products to help you with your environmental monitoring needs. In-Situ Inc. has been designing and manufacturing world-class environmental monitoring instrumentation for over 25 years in the Rocky Mountains of the United States. As it was in the beginning, our expectation is that this product will provide you with many trouble-free years of use. To that end, we pride ourselves on delivering the best customer service and support possible—24 hours a day, 7 days a week. We believe that this level of commitment to you, our customer, is imperative in helping you ensure clean, safe groundwater and surface water resources across the globe. We also understand the need for accurate, reliable assessments and we continue to make significant investments in Research and Development to ensure that we deliver the latest product and technological innovations to support your needs.

Whether you are gathering information about your body of water for a few moments, or over a period of years, you can rely upon us to provide you with a quality product and outstanding customer support at a fair price and have that product delivered to you when and where you need it.

We want your experience with In-Situ Inc. to be pleasant and professional, whether you are renting from us, or purchasing from us. We would be pleased to hear from you and learn more about your needs, and your experiences with our products. Again, we thank you for choosing In-Situ Inc. and we look forward to serving your needs now, and in the future.

Bob Blythe, President and CEO
In-Situ Inc.
bblythe@in-situ.com
WHAT WE PROVIDE

WARRANTY PROVISIONS

In-Situ Inc. warrants the Multi-Parameter TROLL 9500 for one year, RDO sensors for three years, and other sensors for 90 days from date of purchase by the end user against defects in materials and workmanship under normal operating conditions. To exercise this warranty contact Technical Support at the phone or e-mail address listed below for a return material authorization (RMA) and instructions. Complete warranty provisions are posted on our website at www.In-Situ.com.

Maintenance & calibration plans as well as extended warranties are available. Contact your In-Situ representative for complete information.

FIRMWARE & SOFTWARE UPGRADES

From time to time In-Situ may make available enhanced versions of firmware and software to its customers over the Internet. Visit our website at www.in-situ.com to download the latest firmware and software.

HOW TO CONTACT US

Technical Support: 800 446 7488
Toll-free 24 hours a day in the U.S. and Canada

Address: In-Situ Inc.
221 East Lincoln Ave.
Fort Collins, CO 80524
USA

Phone: 970 498 1500
Fax: 970 498 1598
Internet: www.in-situ.com
e-mail: support@in-situ.com

To Obtain Repair Service (U.S.)

If you suspect that your Multi-Parameter TROLL 9500 is malfunctioning and repair is required, you can help assure efficient servicing by following these guidelines:

1. Call or e-mail In-Situ Technical Support (support@in-situ.com). Have the product model and serial number handy.

2. Be prepared to describe the problem, including how the instrument was being used and the conditions noted at the time of the malfunction.

3. If Tech Support determines that service is needed, they will ask that your company pre-approve a specified dollar amount for repair charges. When the pre-approval is received, Tech Support will assign an RMA (Return Material Authorization) number.

TIP: Please keep your RMA number for future reference.

4. Clean the Multi-Parameter TROLL 9500 and cable. Decontaminate thoroughly if it has been used in a toxic or hazardous environment. See the cleaning guidelines and form on the following page.

If an instrument returned for servicing shows evidence of having been used in a toxic or hazardous environment, Customer Service personnel will require written proof of decontamination before they can service the unit.

5. Carefully pack your Multi-Parameter TROLL 9500 in its original shipping box, if possible. Include a statement certifying that the instrument and cable have been decontaminated, and any supporting information.

6. Mark the RMA number clearly on the outside of the box with a marker or label.

7. Send the package, shipping prepaid, to

In-Situ Inc.
ATTN: Repairs
221 East Lincoln Ave.
Fort Collins, CO 80524
USA

The warranty does not cover damage during transit. We recommend the customer insure all shipments. Warranty repairs will be shipped back prepaid.

Outside the U.S.

Contact your international In-Situ distributor for repair and service information.

CERTIFICATION

The MP TROLL 9500 complies with all applicable directives per the CE and FCC and was tested to the EN 61326 / FCC specifications.
CLEANING GUIDELINES

Please help us protect the health and safety of our employees by cleaning and decontaminating equipment that has been subjected to any potential biological or health hazards, and labeling such equipment. Unfortunately, we cannot service your equipment without such notification. Please complete and sign the form below (or a similar statement certifying that the equipment has been cleaned and decontaminated) and send it along to us with each downhole instrument.

- We recommend a good cleaning solution, such as Alconox®, a glassware cleaning product available from In-Situ (Catalog No. 0029810) and laboratory supply houses.
- Clean all cabling. Remove all foreign matter.
- Clean cable connector(s) with a clean, dry cloth. Do not submerge.
- Clean the probe body—including the nosecone, restrictor, cable head, and protective caps. Remove all foreign matter.
- Remove and clean all removable sensors. Rinse with deionized or distilled water after cleaning.

If an instrument is returned to our Service Center for repair or recalibration without a statement that it has been cleaned and decontaminated, or in the opinion of our Service Representatives presents a potential health or biological hazard, we reserve the right to withhold service until proper certification has been obtained.

Alconox is a registered trademark of Alconox Inc.

Decontamination & Cleaning Statement

<table>
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<th>Company Name</th>
<th>Phone</th>
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<th>Instrument Type</th>
<th>Serial Number</th>
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<tr>
<th>Contaminant(s) (if known)</th>
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<tr>
<th>Decontamination procedure(s) used</th>
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<table>
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<tr>
<th>Cleaning verified by</th>
<th>Title</th>
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In-Situ Inc.
SYSTEM DESCRIPTION

The Multi-Parameter TROLL 9500 is a compact, modular system with a range of components to customize the instrument to various applications and operational modes. Components include the instrument body with a host of options, sensors, cables, external power accessories, and consumable items.

STANDARD FEATURES

• 47 mm (1.85 in) diameter
• corrosion-resistant PVC housing with titanium set screws
• 316L stainless steel flow restrictor/sensor board
• Viton® seals
• Fast data sampling
• Continuous real-time readings (profiling)
• User-replaceable off-the-shelf D-cell alkaline batteries, or In-Situ–supplied 3.6V lithium D-cell batteries
• Fully submersible
• RS485 communications
• Low-power microprocessor
• Permanently installed, factory-calibrated temperature sensor
• Permanently installed, factory-calibrated barometric pressure sensor, for use on vented cable
• Temperature-compensated real-time clock
• Easy-to-use Win-Situ® 4 control software for setup, downloading, text and graphical data display
• Optically isolated communication signals
• Cable available in standard and custom lengths of vented or non-vented polyurethane, Halogen-free polyurethane, or Tefzel®
• External power capable

OPTIONAL FEATURES

• Profiler and Professional feature sets, both available in XP with extended parameter capability
• Smart Sensors for water quality measurements—removable, replaceable, field-calibrated
  • Basic Sensors include Dissolved Oxygen (polarographic), pH, combination pH/ORP, Low Conductivity, High Conductivity
  • The Extended Sensor set includes Ammonium, Chloride, Nitrate, Turbidity, RDO® Optical Dissolved Oxygen
• Pocket-Situ full-featured control software for field use on a PDA—take continuous real-time readings, calibrate, program, and download logged data from multiple MP TROLLs
• RuggedReader® handheld PDA
• Temperature-compensated vented or non-vented pressure sensor, available in several ranges, permanently installed and factory-calibrated
• Permanently installed, factory-calibrated turbidity sensor
• Integral data logging capability—up to 4 megabytes flash data storage memory (about a million individual readings)
• Fast data downloads
• Up to 16-test capacity; linear, event, and linear average measurement schedules
• SDI-12 or RS485 interface—or both
• Flow-Sense low-flow sampling software for automated sampling and report creation
• Optional accessories include low-flow flowcell, user-serviceable turbidity wipe, battery-powered magnetic stirrer for use in stagnant water
• Instrument networking and telemetry
CABLES

Several basic cable types are used in the MP TROLL 9500 system:

- **RuggedCable™**, TPU-jacketed (Thermoplastic PolyUrethane)
  - vented or non-vented
  - Halogen-Free vented or non-vented (LSZH-rated, low smoke zero halide)

  *Vented cable is recommended for applications where accurate barometric pressure measurement is required—for example when calibrating dissolved oxygen, or for measuring DO in percent saturation.*

- Vented Tefzel® cable
- Stainless steel suspension wire for use when cable venting is not required (e.g., with an absolute pressure sensor)
- Communication cables for programming the device/downloading the logged data

**RuggedCable™**

Cable includes conductors for power and communication signals, a strength member, and a Kellems® grip to anchor the MP TROLL 9500 securely. Available in standard and custom lengths.

Uphole and downhole ends are identical bayonet-type Twist-Lock connectors that mate with the TROLL 9500 body, TROLL Com communication cable, desiccants, and other accessories.

Vented cable is designed for use with vented pressure/level sensors (gauged measurements). The cable vent tube insures that atmospheric pressure is the reference pressure applied to the pressure sensor diaphragm. Vented cable also enables proper functioning of the internal barometric pressure sensor, and improves dissolved oxygen readings.

Non-vented cable may be used with non-vented pressure/level sensors (absolute measurements), or instruments without a pressure sensor.

* FEP = fluorinated ethylene propylene, the generic equivalent of DuPont Teflon®.
CABLE DESICCANTS

Small Desiccant

Clear cap of indicating silica gel desiccant seals the uphole end of the RuggedCable during use; protects the cable vent tube and device electronics from condensation. The desiccant is blue when active. It will absorb moisture from the top down and for best results should be replaced before the entire volume has turned pink. Replacements are available from In-Situ Inc. or your distributor.

Large Desiccant

The high-volume desiccant pack may last up to 20 times longer than the small desiccant in humid environments. It attaches to the RuggedCable in the same way. Refill kits are available from In-Situ Inc. or your distributor.

COMMUNICATION CABLES

Comm cables interface between the TROLL 9500 and a desktop/laptop PC or handheld PDA for profiling, calibrating, programming, and downloading. Both types include 0.9 m (3 ft) polyurethane cable, external power input jack, and vent with replaceable membrane.

TROLL Com (Cable Connect)

Connects a TROLL 9500’s RuggedCable to a serial or USB port; Weatherproof, withstands a temporary immersion (IP67).

TROLL Com (Direct Connect)

Connects a TROLL 9500 directly to a serial or USB port. A good choice for permanent connection to a PC, or for programming a non-vented TROLL 9500 that will be deployed without RuggedCable. Not designed for field use.

POWER COMPONENTS

The MP TROLL 9500 operates in 2 power modes
- internal power
- external AC line power

INTERNAL POWER

The MP TROLL 9500 uses:
- two standard 1.5V alkaline D cells, OR
- two 3.6V lithium D cells—recommended for use with an RDO optical dissolved oxygen sensor, and with a turbidity wiper

Use only Saft LSH-20 3.6V lithium D cells. Use of any other lithium battery will void the product warranty.

EXTERNAL POWER

A single MP TROLL 9500 can run exclusively on power supplied from a 9-12 VDC line power supply connected to a 90-264 VAC input. When line power is enabled, the TROLL shuts down the battery regulator, thus preserving the internal batteries. All TROLL Com models include an external power input jack.

Communication Cables

- RS232 TROLL Com, Cable Connect ...................................... 0056140
- RS232 TROLL Com, Direct Connect ................................. 0056150
- USB TROLL Com, Cable Connect ................................. 0052500
- USB TROLL Com, Direct Connect ................................. 0052510

Accessories

- Catalog No.
- Accessory
- Small desiccant (3) ................................................................. 0052230
- Large desiccant, plastic connector ........................................ 0053550
- Large desiccant, titanium connector .................................... 0051810
- Large desiccant refill kit ....................................................... 0029140

Power Components

- Catalog No.
- Accessory
- AC Adapter 9V ................................................................. 0031880
- Replacement batteries, alkaline (1) .................................. 0042020
- Lithium battery (2) kit ......................................................... 0048230

TIP: Battery life is dependent upon temperature, cable length, and how often the device is recording measurements.
### Section 2: Components & Features

**WATER QUALITY ACCESSORIES**

<table>
<thead>
<tr>
<th>Catalog No.</th>
<th>Description</th>
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</thead>
<tbody>
<tr>
<td>0032110</td>
<td>Dissolved Oxygen: 1 liter DI water, 500 mL Na₂SO₃</td>
</tr>
<tr>
<td>0033250</td>
<td>Quick-Cal: 4 x 250 mL (for calibrating Basic Sensors)</td>
</tr>
<tr>
<td>0042210</td>
<td>Battery-Powered Magnetic Stirrer</td>
</tr>
<tr>
<td>0044510</td>
<td>Turbidity Wiper</td>
</tr>
<tr>
<td>0048230</td>
<td>Lithium battery kit (2 “D” cells, 3.6V ea.)</td>
</tr>
<tr>
<td>0048580</td>
<td>RDO bubbler cal kit for 0084310 sensor</td>
</tr>
<tr>
<td>0048590</td>
<td>RDO bubbler cal kit for 0085070 sensor</td>
</tr>
<tr>
<td>0044520</td>
<td>Replacement wiper head</td>
</tr>
<tr>
<td>0044530</td>
<td>Wiper pad replacement kit</td>
</tr>
<tr>
<td>0045510</td>
<td>Calibration Kits (four liters unless otherwise noted)</td>
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<thead>
<tr>
<th>Catalog No.</th>
<th>Description</th>
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<tbody>
<tr>
<td>0032050</td>
<td>Nitrate</td>
</tr>
<tr>
<td>0032060</td>
<td>Ammonium</td>
</tr>
<tr>
<td>0032070</td>
<td>Chloride</td>
</tr>
<tr>
<td>0032870</td>
<td>RDO Optical Dissolved Oxygen sensor</td>
</tr>
<tr>
<td>0032080</td>
<td>RDO Optical Dissolved Oxygen sensor for use with pressure and/or turbidity sensor, sub-2”</td>
</tr>
<tr>
<td>0032090</td>
<td>RDO Optical Dissolved Oxygen sensor for use with pressure and/or turbidity sensor, sub-4”</td>
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<tr>
<th>Catalog No.</th>
<th>Description</th>
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<tbody>
<tr>
<td>0032120</td>
<td>pH: 1 each pH 4, pH 7, pH 10, DI water</td>
</tr>
<tr>
<td>0032130</td>
<td>pH/ORP: 1 each pH 4, pH 7, pH 10, DI water, 1 liter Zobell’s Solution</td>
</tr>
<tr>
<td>0032140</td>
<td>Ammonium: 1 each 14 ppm, 140 ppm, 1400 ppm, DI water</td>
</tr>
<tr>
<td>0032150</td>
<td>Chloride: 1 each 35.5, 355, 3545 ppm, DI water</td>
</tr>
<tr>
<td>0032160</td>
<td>Low Chloride: 2 each 35.5, 355 ppm</td>
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<thead>
<tr>
<th>Catalog No.</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>0032170</td>
<td>High Nitrate: 2 each 140, 1400 ppm</td>
</tr>
<tr>
<td>0032180</td>
<td>Nitrate: 1 each 14 ppm, 140 ppm, 1400 ppm, DI water</td>
</tr>
<tr>
<td>0032190</td>
<td>Low Ammonium: 2 each 14, 140 ppm</td>
</tr>
<tr>
<td>0032200</td>
<td>High Chloride: 2 each 355, 3545 ppm</td>
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<tr>
<td>0032300</td>
<td>Chloride: 1 each 255 ppm, 3545 ppm</td>
</tr>
<tr>
<td>0032310</td>
<td>Chloride: 1 each 35.5 ppm, 355 ppm</td>
</tr>
<tr>
<td>0032320</td>
<td>Chloride: 1 each 35.5 ppm, 355 ppm, DI water</td>
</tr>
<tr>
<td>0032330</td>
<td>High Chloride: 2 each 355, 3545 ppm</td>
</tr>
<tr>
<td>0032340</td>
<td>Low Chloride: 2 each 35.5, 355 ppm</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Catalog No.</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>0032350</td>
<td>Nitrate: 1 each 14 ppm, 140 ppm, 1400 ppm, DI water</td>
</tr>
<tr>
<td>0032360</td>
<td>Low Nitrate: 2 each 14, 140 ppm</td>
</tr>
<tr>
<td>0032370</td>
<td>High Nitrate: 2 each 140, 1400 ppm</td>
</tr>
<tr>
<td>0032380</td>
<td>Nitrate: 1 each 14 ppm, 140 ppm, 1400 ppm, DI water</td>
</tr>
<tr>
<td>0032390</td>
<td>Low Ammonium: 2 each 14, 140 ppm</td>
</tr>
<tr>
<td>0032400</td>
<td>High Chloride: 2 each 355, 3545 ppm</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Catalog No.</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>0032410</td>
<td>Nitrate: 1 each 14 ppm, 140 ppm, 1400 ppm, DI water</td>
</tr>
<tr>
<td>0032420</td>
<td>Low Nitrate: 2 each 14, 140 ppm</td>
</tr>
<tr>
<td>0032430</td>
<td>High Nitrate: 2 each 140, 1400 ppm</td>
</tr>
<tr>
<td>0032440</td>
<td>Nitrate: 1 each 14 ppm, 140 ppm, 1400 ppm, DI water</td>
</tr>
<tr>
<td>0032450</td>
<td>Low Ammonium: 2 each 14, 140 ppm</td>
</tr>
<tr>
<td>0032460</td>
<td>High Chloride: 2 each 355, 3545 ppm</td>
</tr>
</tbody>
</table>

### Calibration Solutions

**Individual Calibration Solutions (one liter unless otherwise noted)**

<table>
<thead>
<tr>
<th>Catalog No.</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>0032670</td>
<td>Calibration Solution: 0.17767 g/t Na₂SO₃ (Sodium Sulfite) for DO Cal, 500 mL</td>
</tr>
</tbody>
</table>
| 0032560     | Conductivity, 147 µS  
| 0032570     | Conductivity, 12890 µS  
| 0032580     | Conductivity, 58670 µS  
| 0032590     | pH 4  
| 0032600     | pH 7  
| 0032610     | pH 10  
| 0032620     | ZoBell’s Solution (ORP)  
| 0032630     | Nitrate, 14 ppm as N  
| 0032640     | Nitrate, 1400 ppm as N  
| 0032650     | Ammonium, 14 ppm as N  
| 0032660     | Ammonium, 1400 ppm as N  
| 0032670     | Chloride, 35.5 ppm  
| 0032680     | Chloride, 3545 ppm  
| 0032690     | Chloride, 35.5 ppm, 355 ppm, DI water  
| 0032700     | Chloride: 2 each 355, 3545 ppm  

### Maintenance/Service/Replacement Parts

<table>
<thead>
<tr>
<th>Catalog No.</th>
<th>Description</th>
</tr>
</thead>
</table>
| 0032710     | Chloride: 2 each 355, 3545 ppm  
| 0032720     | Chloride: 355 ppm  
| 0032730     | Chloride: 3545 ppm  
| 0032740     | Turbidity, 10 NTU polymer suspension, 500 mL  
| 0032750     | Turbidity, 100 NTU polymer suspension, 500 mL  
| 0032760     | Turbidity, 1000 NTU polymer suspension, 500 mL  
| 0032770     | Turbidity, 1800 NTU polymer suspension, 500 mL  
| 0032780     | Sensor insertion tool  
| 0032790     | Sensor removal tool  
| 0032800     | Cal cup, PVC  
| 0032810     | Replacement wiper head  
| 0032820     | Wiper pad replacement kit  
| 0032830     | Lithium battery kit (2 “D” cells, 3.6V ea.)  

**TROLL 9500 Operator’s Manual**

8 0095110 rev. 007 01/09
MP TROLL 9500 Maintenance kit (o-rings, lubricant, dust caps, sensor port plugs) .............................................................. 0095100
MP TROLL 9500 Maintenance kit (o-rings, lubricant, dust caps, sensor port plugs) for use with 0085070 RDO sensors ...... 0095100
DO sensor service kit, 1-mil membranes ....................................................... 0033200
DO sensor service kit, 2-mil membranes ....................................................... 0033440
RDO Sensor Cap replacement kit ................................................................. 0084230
RDO Maintenance kit (sensors purchased before 09/08) ........................... 0048250
RDO Foil replacement kit (sensors purch. before 09/08) ......................... 0048500
pH & pH/ORP Reference Filling Solution (60 mL) ...................................... 0056900
pH & pH/ORP Reference Junction kit ........................................................... 0059620
pH & pH/ORP Sensor Storage Solution (500 mL) ......................................... 0065370

INSTALLATION ACCESSORIES

- Twist-Lock Hanger: stainless steel hanger to suspend a non-vented TROLL 9500, Level TROLL, or Baro TROLL while taking data; no venting, no communication capabilities
- Cable Extender: connects two lengths of RuggedCable
- Wellcaps, locking and vented
- Well Docks: top-of-well support for 2", 4", or 6" well
- SDI-12 adapter: power and signal management for SDI-12 communication

Accessory                        Catalog No.
Twist-Lock Hanger                0051480
Cable Extender                   0051490
Locking Wellcap, 2"               0020360
Locking Wellcap, 2" vented       0020370
Locking Wellcap, 4"               0020380
Locking Wellcap, 4" vented       0020390
Top-of-well installation ring     WELLDOCK2", 4", 6" 0095200, 0095210
SDI-12 adapter                   0095200, 0095210
Flow Cell (sub-2 in)             0044710
Flow Cell (sub-4 in)             0057600

CONTROL SOFTWARE

Win-Situ® 4 enables communication between the MP TROLL 9500 and a desktop or laptop PC.

Win-Situ provides instrument control for calibration, profiling, direct readings, data logging, data extraction, data viewing (text and graphical interface), choice of units and other custom display options, battery/memory usage tracking, interface to networks and telemetry.

System requirements: Microsoft® Windows® 2000, Windows XP, or Windows Vista™; Internet Explorer (IE) 5.0 or later, and a CD-ROM drive.

Pocket-Situ 4 provides Win-Situ’s features and functions on a field-portable platform. Requirements: In-Situ RuggedReader® or other supported PDA running Microsoft Pocket PC (Windows Mobile®) 2003 or later, with a serial communications port for connection to the TROLL 9500, and at least 16 megabyte capacity for data storage (SD card, CF card, or the device’s built-in non-volatile memory).

For installation and file exchange, Pocket-Situ requires the following installed on an office desktop or laptop computer:

- Microsoft® ActiveSync®
- Win-Situ 4
- Optional: Win-Situ Sync (or Pocket-Sync 4, earlier version of Win-Situ Sync)
**PRODUCT SPECIFICATIONS**

**Wetted Materials**
- PVC, titanium, Viton®, acetal, 316L SS

**Operating Temperature**
- -5°C to 50°C (23°F to 122°F)

**Storage Temperature**
- -40°C to 80°C (-40°F to 176°F)

**Pressure Rating**
- 350 psi (246 m, 807 ft)

**Dimensions**
- 47 mm (1.85 in) dia, x 57.7 cm (22.7 in) long
  - with RDO adapter: 88.4 mm (3.48 in) dia, 57.7 cm (22.7 in) long

**Weight (without cable)**
- 1.4 Kg (3.1 lbs)

**Power**
- Battery type: 2 standard alkaline D-cells (1.5 V), or 2 lithium D cells (approx. 3.6 V)
- External power input: 9-12 VDC (optional)

**Data Sampling**
- Memory type/size (memory equipped models): 4 megabytes flash data storage, about 1,000,000 individual readings
- Fastest linear logging rate: 5 seconds (10 seconds with RDO)
- Profiling speed: 2 seconds

**Communications**
- RS485

**Computer interface**
- RS232

**Software**
- Win-Situ® 4: Desktop/laptop PC with Microsoft® Windows® 2000 SP2 or later (Windows XP, Windows Vista™), Internet Explorer 5.0 or later, serial com port, CD-ROM drive, 16-64 Mb RAM
- Pocket-Situ 4: In-Situ RuggedReader® or other supported handheld PDA running Microsoft Pocket PC (Windows Mobile®) 2003 or later

**Cable**
- Jacket options: Polyurethane, halogen-free (HF) polyurethane, Tefzel®
- Conductors: 6 conductors, 24 AWG, polypropylene insulation
- Diameter: 6.7 mm (0.265 in)
- Connector: Titanium, 18.5 mm (0.73 in) O.D.
- Break strength: 127 kg (280 lb)
- Minimum bend radius: 2X cable diameter (13.5 mm, 0.54 in)
- Weight: Vented, regular & HF: 14 kg/300 m (32.3 lb/1000 ft)
  - Non-vented, regular & HF: 16 kg/300 m (35.6 lb/1000 ft)
  - Vented Tefzel®: 23kg/300m (52 lb/1000 ft)

**Factory-Installed Sensors**

<table>
<thead>
<tr>
<th>Pressure/level</th>
<th>Type</th>
<th>Range</th>
<th>Accuracy</th>
<th>Resolution</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pressure/level</td>
<td>Media-isolated piezoresistive silicon strain gauge</td>
<td>15 psig (0-11 m, 0-35 ft)</td>
<td>± 0.1% FS</td>
<td>1 mm</td>
</tr>
<tr>
<td>Temperature</td>
<td>Platinum resistance thermometer</td>
<td>-5°C to 50°C (23°F to 122°F)</td>
<td>± 0.1°C</td>
<td>0.01°C</td>
</tr>
<tr>
<td>Barometric pressure</td>
<td>Piezoresistive silicon pressure sensor</td>
<td>0-16.5 psia (854 mm Hg, 33.6 in Hg)</td>
<td>± 0.3% FS (2.54 mm Hg, 0.1 in Hg)</td>
<td>0.1 NTU</td>
</tr>
<tr>
<td>Turbidity Wiper</td>
<td>Nephelometer, 90° light scattering, 870 nm LED, solid-state</td>
<td>0-2000 NTU</td>
<td>± 5% or 2 NTU (whichever is greater)</td>
<td>0.1 NTU</td>
</tr>
</tbody>
</table>

**Basic Sensors**

<table>
<thead>
<tr>
<th>Conductivity</th>
<th>Type</th>
<th>Operating Range</th>
<th>Accuracy</th>
<th>Resolution</th>
</tr>
</thead>
<tbody>
<tr>
<td>Conductivity</td>
<td>4-cell conductivity, AC drive</td>
<td>Low Range sensor: 5 µS/cm to 20,000 µS/cm</td>
<td>± 0.5% or 2 µS/cm (whichever is greater) when calibrated in region of interest.</td>
<td>Range-dependent</td>
</tr>
<tr>
<td>High Range sensor: 150 µS/cm to 112,000 µS/cm</td>
<td>± 0.5% or 2 µS/cm between 150 µS/cm and 112,000 µS/cm when calibrated in region of interest. From 70 to 150 µS/cm &amp; 112,000 to 200,000 µS/cm ± 2% + 4 µS/cm typical</td>
<td>350 psi (246 m, 807 ft)</td>
<td>0°C to 50°C</td>
<td></td>
</tr>
</tbody>
</table>

---

* Base unit = MP TROLL 9500 & factory-installed sensors

---

**SECTION 2: COMPONENTS & FEATURES**
### pH

**Type**  
Glass sensing bulb, single-junction electrode, replaceable ceramic junction, refillable reference electrolyte

**Range**  
0 to 12 pH units

**Pressure Rating**  
350 psi 246 m, 807 ft

**Operating Temp.**  
0°C to 50°C (32°F to 122°F)

**Accuracy**  
± 0.1 pH unit

**Resolution**  
0.01 pH unit

### pH/ORP

**Type**  
Glass sensing bulb, platinum wire, single-junction electrode, replaceable ceramic junction, refillable reference electrolyte

**Range**  
± 1400 mV, 0 to 12 pH

**Pressure Rating**  
350 psi 246 m, 807 ft

**Operating Temp.**  
0°C to 50°C (32°F to 122°F)

**Accuracy**  
± 4 mV, ± 0.1 pH unit

**Resolution**  
1 mV, 0.01 pH unit

### Dissolved Oxygen (polarographic)

**Type**  
Clark polarographic

**Range**  
0 to 20 mg/L, 0 to 200% saturation

**Pressure Rating**  
350 psi (246 m, 807 ft); submersion & retrieval at up to 4 ft per second

**Operating Temp.**  
-5°C to 50°C (23°F to 122°F)

**Accuracy**  
± 0.2 mg/L

**Resolution**  
0.01 mg/L

### Ammonium (NH₄⁺)

**Type**  
PVC membrane sensing element, double junction Ag/AgCl reference half-cell, reference electrolyte gel

**Range**  
0.14 to 14,000 ppm N (0.1 to 18,000 ppm NH₄⁺)

**Pressure Rating**  
20 psi (14 m, 46 ft)

**Operating Temp.**  
-5°C to 40°C (23°F to 104°F)

**Accuracy**  
± 10%

**Resolution**  
0.01 ppm

### Chloride (Cl⁻)

**Type**  
Solid-state sensing electrode, double junction Ag/AgCl reference half-cell, reference electrolyte gel

**Range**  
0.35 to 35,500 ppm Cl (2 to 35,000 ppm Cl⁻)

**Pressure Rating**  
100 psi (70 m, 231 ft)

**Operating Temp.**  
-5°C to 50°C (23°F to 122°F)

**Accuracy**  
± 15%

**Resolution**  
0.01 ppm

### Nitrate (NO₃⁻)

**Type**  
PVC membrane sensing element, double junction Ag/AgCl reference half-cell, reference electrolyte gel

**Range**  
0.14 to 14,000 ppm N (0.4 to 62,000 ppm NO₃⁻)

**Pressure Rating**  
20 psi (14 m, 46 ft)

**Operating Temp.**  
-5°C to 40°C (23°F to 104°F)

**Accuracy**  
± 10%

**Resolution**  
0.01 ppm

### Extended Sensors

### Dissolved Oxygen (optical, RDO)

**Type**  
Optical, fluorescence quenching

**Range**  
0 to 20 mg/L, 0 to 450% saturation

**Pressure Rating**  
300 psi

**Operating Temp.**  
0°C to 40°C (32°F to 104°F)

**Accuracy**  
± 0.1 mg/L @ 0-8 mg/L  
± 0.2 mg/L @ 8-20 mg/L

**Resolution**  
0.01 mg/L

### Typical Battery Life @ 20°C

<table>
<thead>
<tr>
<th>Battery type</th>
<th>Installed sensors</th>
<th>Sample Interval</th>
</tr>
</thead>
<tbody>
<tr>
<td>2 alkaline D-cells</td>
<td>Pressure, temperature, baro, pH/ORP, conductivity, polarographic (Clark) D.O. All of the above plus turbidity All of the above plus turbidity &amp; wiper Pressure, temp., baro, pH/ORP, conductivity, RDO, turbidity</td>
<td>60 min. 76 days 75 days 63 days</td>
</tr>
<tr>
<td>2 lithium D-cells</td>
<td>Pressure, temperature, baro, pH/ORP, conductivity, RDO, turbidity, no wiper</td>
<td>270 days</td>
</tr>
</tbody>
</table>
This section provides a quick overview of the initial steps necessary to get the Multi-Parameter TROLL 9500 ready to take measurements.

You will need—

- Power
- Sensors
- Software
- Cable
  TROLL Com communication cable for deployment
- Calibration Kit—one for each sensor to be calibrated, or the QuickCal Kit for the Basic Sensor set (pH, ORP, polarographic D.O., Conductivity)

**Provide Power**

*TIP:* When the unit ships with a Polarographic Dissolved Oxygen sensor installed, alkaline batteries are pre-installed in the device to power the D.O. sensor and keep it conditioned.

Install batteries in the Multi-Parameter TROLL 9500 as follows:

1. Unscrew and remove the battery compartment cover. Slide it up over the cable (if attached).

2. Insert two D-size batteries negative side first, positive side up.

3. Replace the battery compartment cover and tighten to compress the o-ring seals.

*TIP:* The MP TROLL 9500 uses standard off-the-shelf 1.5V alkaline D-cells.

*Alternatively, you may use Saft LSH-20 3.6V lithium D cells. Use of any other lithium battery will void the product warranty.*
**INSTALL SENSORS**

Basic water-quality sensors—pH or pH/ORP, polarographic D.O., Conductivity—may arrive installed in the instrument’s sensor ports. Proceed to **Install Software** if there are no additional sensors to be installed.

Install sensors in the Extended Sensor Set and any sensors shipped separately as follows:

**TIP:** The RDO® Optical Dissolved Oxygen sensor has special installation requirements. Please refer to the RDO Quick Start guide, or see Section 13 of this operator’s manual for complete information.

1. Remove the restrictor or Cal Cup from the front end of the MP TROLL 9500.

   ![Sensor Block Diagram](Image)

   This allows access to the sensor block depicted in the drawing below.

   There are four sensor ports, 1 – 4, plus a permanently installed pressure and/or turbidity sensor or plug.

2. Remove the sensor’s protective cap or storage bottle and set aside for future storage of the sensor. If the connector end is covered with a cap, remove it also.

3. Remove any moisture or dirt from the area around the port where you will install the sensor, then use the sensor removal tool to remove the plug from the port. Retain the plug for use when with fewer than 4 removable sensors installed.

4. Remove any moisture or dirt from the port connector with a clean swab or tissue.

5. Check lubrication of the sensor o-rings.

   **Warning:** The sensor o-rings require generous lubrication before installation. New sensors will be lubricated at the factory. If the o-rings appear dry, apply a silicone lubricant before installation.

6. Align the mark on the side of the sensor with the mark on the correct port (see diagram), or visually align the sensor connector pins with the port connector pins.

   **Caution:** If the sensing element is at the end of the sensor, be careful to handle the sensor by the sides. Use the insertion tool to press the sensor into the port at step 7.

7. Press the sensor firmly into the port until you feel it dock with the connector at the bottom.

   When the sensor is properly inserted, a small gap (the width of the sensor removal tool) remains between the instrument body and the widest part of the sensor, for ease of removal.

**Warning:** After installing a polarographic D.O. sensor, we recommend conditioning it for 2-4 hours, preferably overnight, before calibrating. For more on D.O. sensor conditioning, see Section 13.

---

**END VIEW OF SENSOR BLOCK**

- **D.O. (polarographic) or ammonium or chloride or nitrate**
- **Pressure/Turbidity (or plug)**
- **Conductivity**
- **Temperature**
- **Alignment mark**
- **RDO or pH or ammonium or chloride or nitrate or turbidity wiper**
- **pH/ORP or RDO or pH or ammonium or chloride or nitrate**

---

**END VIEW WITH THE RDO CABLE CONNECT SENSOR PLUGGED INTO PORTS 1 OR 3**

**END VIEW WITH THE RDO DIRECT CONNECT SENSOR PLUGGED INTO PORTS 3 AND COVERING PORTS 2 AND 5.**
INSTALL THE SOFTWARE

WIN-SITU 4
Install Win-Situ 4 from the In-Situ software CD or the In-Situ website:
• Click on Win-Situ 4 and follow the instructions to install Win-Situ 4 to your local hard drive.

USB TROLL COM DRIVERS
• Select the option to install USB TROLL Com drivers during the Win-Situ installation. Two drivers will be loaded to your hard drive, (USB TROLL Com, USB TROLL Com serial port).

POCKET-SITU 4
For communication using a RuggedReader® handheld in the field, install the desktop component of Pocket-Situ 4 from the CD or website. The desktop component is the “Win-Situ Software Manager,” and helps you install Pocket-Situ on the RuggedReader at any time.
• Click on Pocket-Situ 4 and follow the instructions to install the Win-Situ Software Manager to your local hard drive.

To install Pocket-Situ on the RuggedReader: When convenient, connect the RuggedReader to the PC, establish a connection in Microsoft ActiveSync®, launch the Win-Situ Software Manager, and follow the instructions.

WIN-SITU SYNC
If you plan to synchronize data files from the RuggedReader back to a desktop PC after collecting data in the field, also install Win-Situ Sync from the CD or website.

CONNECT THE HARDWARE

TROLL 9500 TO TROLL COM
Connect the TROLL 9500 to the appropriate TROLL Com as shown.
• Connect a Direct Connect model simply by pressing onto the connector at the back end of the TROLL 9500.
• Attach a Cable Connect model via locking Twist-Lock connector to the TROLL’s RuggedCable—see the box on the next page.

TROLL COM TO COMPUTER
Plug an RS232 TROLL Com into a serial port on a desktop/laptop PC or a RuggedReader.
Plug a USB into a USB port on a desktop/laptop PC. Install drivers and check the virtual COM port—see the box below.

After connections are made, you are ready to launch the software and program the MP TROLL 9500.

RS232 connections
USB connections

REMEMBER THE COM PORT NUMBER! You will need it when connecting to the TROLL 9500 in software.
**TWIST-LOCK CABLE CONNECTIONS**

RuggedCable mates to the TROLL 9500 and to Cable Connect TROLL Coms with a bayonet-type Twist-Lock connector.

1. Remove the soft protective caps from the TROLL 9500 and the cable.

   ![Image of TROLL 9500 and Cable]

   **TIP:** Keep the dust caps to protect the connector pins and O-ring when the connectors are not mated.

2. Take a moment to look at the connectors. Each has a flat side. Note the pins on the body connector (one on each side) and the diagonal slots on the cable connector (one on each side).

3. Slide back the sleeve on the cable connector.

4. Orient the flat sides so they will mate up, and insert the body connector firmly into the cable connector.

5. Slide the sleeve on the cable toward the body until the pin on the body pops into the round hole in the slot on the cable.

6. Grasp the textured section of the cable connector in one hand and the body in the other. Push and twist firmly so that the pin on the body slides along the slot on the cable and locks securely into the other hole. The “click” ensures the cable is securely attached.

To attach a Cable Connect TROLL Com, first remove the desiccant from the cable (if present): Grasp the textured section of the cable connector in one hand and the desiccant in the other. Twist in opposite directions to unlock the desiccant from the cable.

Orient the “flats” so they will mate up, and insert the TROLL Com connector firmly into the cable connector.

Push, twist, and click to lock.
ESTABLISH COMMUNICATION WITH THE MP TROLL 9500

1. Start Win-Situ 4 by double-clicking the shortcut created on the desktop during installation.
   
   Or tap the Pocket-Situ 4 shortcut on the PDA Start Menu.

2. The Connection Wizard starts to help you set up the port:
   - **Connection type:**
     - Direct, for use when the Multi-Parameter TROLL 9500 is attached directly to the host PC
     - Modem for a dial-up modem
     - Spread-Spectrum Radio
   - **Devices connected:** Select “one” to speed up connection to a single device, or “more than one” for a network.
   - **Port:** Select the COM port to which the device is attached. If using a USB TROLL Com, be sure to select the correct COM port (see the box on page 14). For a Modem connection, enter the phone number—up to 40 characters; a comma designates a pause.
   - **Baud Rate:** Select any rate (19,200 is the default); baud rates are automatically synchronized between devices and the host PC.
   - **Name (optional):** A default connection name is suggested.
   - To have the wizard connect automatically to the attached device(s), be sure the option **Connect and find devices on “Finish”** is checked.

3. When you finish the Wizard, the software “finds” (connects to) the device, and displays it in the Navigation tree.

   **TIP:** For an introduction to the appearance and controls of the user interface, turn to Section 4, Control Software.

SET THE REAL-TIME CLOCK

Data collection schedules depend on the device’s real-time clock, shown in the lower right corner of the desktop PC interface. If the device clock is wrong, be sure to correct it before scheduling tests. Set the clock as follows:

1. With the device selected in the Navigation tree, press **Edit**.

2. In the Device Wizard, select **Clock**.

3. Follow the instructions to synchronize the MP TROLL 9500’s internal real-time clock to the host computer.

4. When you finish the Wizard, Win-Situ sends the information to the device and updates the display.

   **TIP:** Due to the size of the PDA screen, the device clock is not displayed in the Pocket-Situ interface. We recommend you edit the device—Tap the device, tap Edit, tap Clock—to synchronize the device clock to the PDA clock.

---

**Q:** What do I do if the MP TROLL 9500 does not appear in the Navigation tree?

**A:** First try double-clicking the connection. If the device still does not appear, there may be a communication problem. Check these likely culprits:
- Be sure you selected the correct COM port for your PC.
- Check that all cable connections are tight.
- Be sure the MP TROLL 9500 has sufficient battery power, or that external power is being supplied.
QUICK-CAL THE BASIC WATER-QUALITY SENSORS

You will need:

- MP TROLL 9500 with Basic sensors installed (pH or pH/ORP, polarographic D.O., Conductivity), plugs in any unused ports.
- Cal Cup. Remove sponge if present.
- Quick Cal solution, at room temperature. Shake well before use.

1. Fill the Cal Cup with the Quick Cal solution. See guidelines in the sidebar on this page.

2. Remove the restrictor (if attached) and insert the front end of the MP TROLL 9500 into the Cal Cup. Thread the Cal Cup onto the body until seated against the o-ring, then back off slightly to avoid overtightening.

3. Connect the MP TROLL 9500 to a PC and establish a connection in Win-Situ 4 or Pocket-Situ 4.

4. Select the MP TROLL 9500 in the Navigation tree. The software will automatically detect and display the installed sensors.

5. In the Navigation tree, click on Parameters.

6. In the Information pane, select QuickCal.

Quick-Cal Tips

- A Quick Cal is the fastest way to calibrate pH, conductivity, polarographic D.O., & ORP. But for best results, if your software supports it, we recommend traditional calibration for pH, conductivity, and D.O., as described in Sections 11-13.

- Be sure to condition a new polarographic D.O. sensor for 2-4 hours, preferably overnight, before calibrating. See Section 13 for information. If the sensor is installed when you receive the instrument, it is conditioned and ready to calibrate.

- Guidelines for filling the Cal Cup with Quick Cal solution:
  - With a full complement of sensors installed, use the lower line as a guide (about half full).
  - With 1 or 2 sensors installed, fill to the upper line.
  - When calibrating a high-range conductivity sensor, insure the side ports are immersed in solution. Dislodge any bubbles that may appear. For a Quick Cal with a full set of sensors, we suggest you fill the Cal Cup above the line labeled “Recommended Fill Line - High.” Use about a half-inch of additional fluid, depending on the sensor load. Some experimentation may be needed.

- When Quick-Calibrating polarographic D.O. with a high-range conductivity sensor installed, the increased fill level suggests handling with care when you invert the Cal Cup at step 11—allow the excess fluid to spill into a sink, or remove the Cal Cup from the instrument and pour some fluid out, re-attach the Cal Cup, then invert it and loosen the end cap to achieve the correct atmospheric pressure for a polarographic D.O. calibration. The D.O. sensor membrane should be in air, the temperature sensor should be completely submerged.

- For best accuracy, conductivity sensors should be wetted for 15-30 minutes immediately prior to calibration. This immersion can be in either clean water or the Quick Cal solution.

- For best results at temperatures at or below 15°C, de-select the conductivity sensor when performing a Quick-Cal; follow up with a single-parameter calibration using 8000 microSiemens/cm.

- If an RDO Optical Dissolved Oxygen sensor is installed, use the special soft plastic cal insert (it has an orange base) to Quick Cal the standard sensors, since the standard Cal Cup will not fit when the RDO sensor is installed. Fill the cal insert with calibration solution, slide it up around the sensors, including the RDO cable, and use the RDO restractor as a support during calibration.

- If your MP TROLL 9500 includes a turbidity wiper, insure the pad does not absorb Quick Cal solution. Either carefully remove the wiper head (see Wiper Maintenance in Section 18), or soak the front end of the TROLL in water before calibrating; allow the pad to become saturated.

- Quick Cal is not available for these sensors: Nitrate, Ammonium, Chloride, Turbidity, Optical D.O. Traditional calibrations should be performed for best results from these sensors.
The Quick Calibration Wizard starts. A screen like this is displayed.

7. Insure there is a check beside the sensors you wish to Quick-Cal.

8. Click Next. The pH, ORP, and Conductivity screen appears.

9. Click Run to start the calibration.

The display will continuously update as readings are taken and compared against the stabilization criteria (see sidebar).

All 3 parameters must indicate NOMINAL or STABLE before the calibration can continue.

- When pH, ORP, and conductivity are STABLE, the polarographic D.O. calibration screen is displayed automatically if a polarographic D.O. sensor is installed.

### Status indicators during stabilization:

**NOT TESTED** may be displayed before the calibration begins.

**UNSTABLE** indicates the sensor response does not meet the criteria for a valid calibration point. All parameters start out at “Unstable” status.

**NOMINAL** indicates the change in the sensor response over time meets a relaxed stability criterion. “Nominal” stability will occur first, and can be accepted to shorten the calibration time.

**STABLE** indicates the change in the sensor response over time meets the stability criteria for a Quick Cal.

**Reading**: Current sensor response (in indicated units).

**Deviation**: Change in response between the last two readings. This enables you to follow the progress of the stabilization, but deviation from the previous reading is not necessarily the best indicator of stability as the software is looking at longer-term trends.

- **Run** button: Starts the calibration
- **Accept** button: Becomes available when Nominal stability is reached (see below)
- **Stop** button: Stops the current phase of the calibration
- **Back** button: Goes to the previous screen (when available)
- **Next** button: Proceeds to the next screen (when available)
- **Cancel** button: Cancels the calibration

### What is the difference between NOMINAL and STABLE?

To meet the criteria for a valid calibration point, the change (deviation) in sensor response is monitored over time. The software is looking for the calibration solution temperature and the sensor readings to settle over a specific time period. The criteria for **STABLE** are designed to meet the published specifications. The **NOMINAL** criteria are designed to shorten the calibration time when an approximate calibration is acceptable. When the deviation falls within the limits of the “loosened” specifications, **NOMINAL** is displayed in the Status area, and the Accept button becomes available to store the current calibration point.

Accepting a NOMINAL value may save considerable time. In some cases, especially if the sensors have been soaking in the solution for several minutes prior to calibration, the accuracy achieved by accepting a nominal value may be very similar to that obtained by waiting for complete stability.
Alternatively, you may click Accept to store the early values. The Accept button becomes available when NOMINAL or STABLE is indicated for all 3 parameters.

If there is no polarographic D.O. sensor, the Quick Cal is complete. Go to step 14.

If performing a Polarographic Dissolved Oxygen calibration, continue with steps 10-13.

10. Polarographic Dissolved Oxygen: Before the D.O. segment of the Quick Cal Wizard starts, you will be asked how you want to handle barometric pressure.

Barometric pressure is important in converting measurement of D.O. concentration to percent saturation, and a value is required for accurate calibration. If the TROLL 9500 cable is vented, an accurate barometric pressure value can be read from the onboard barometric pressure sensor. If the TROLL cable is non-vented, then a barometric pressure value should be entered manually.

Do one of the following in this screen:

- If the TROLL 9500 is on vented cable now and will take measurements using vented cable, click No — and you may want to check the “Don’t ask me this again” box.
- If the device is on vented cable now but will take measurements on non-vented cable, click Yes. In the Edit Barometric Channel screen, check the box indicating non-vented cable for measurements but vented cable for calibration/programming.
- If the device is on non-vented cable now and will take measurements on non-vented cable, click Yes. In the Edit Barometric Channel screen, check the box indicating non-vented cable for measurements and enter a barometric pressure value. For help, see Section 9, Monitoring Barometric Pressure.

11. To complete the calibration, expose the polarographic D.O. sensor to air: Without disconnecting the cable, invert the probe so that the membrane at the tip of the sensor is in air and the temperature sensor is immersed in fluid. This will probably require you to remove the end cap from the Cal Cup and pour some fluid out.

While you have the cap off, remove any droplets on the membrane by blotting gently with a clean swab or the corner of a tissue. Re-attach the end cap loosely to insure the volume is not pressurized.

TIP: For proper venting, loosen the end cap until a small hole in the threads near the o-ring is at least partly visible.

12. In the DO calibration screen, select the membrane type, stamped on the membrane module (if not indicated, select 1-mil Teflon).

13. Click Run to begin the stabilization.

The display will continuously update as readings are taken and compared against the stabilization criteria (see sidebar on the previous page).

14. When the sensor response is STABLE (or alternatively, when you accept the nominal value), the Quick Cal procedure is complete.
The Wizard displays a final calibration screen like the one below at the end of the Quick Calibration procedure. This information is also stored in the Calibration Report. Click Finish to continue. When you click Finish, the calibration information is written to the sensors.

The calibrated sensors are ready to take measurements.

Ideally, you should calibrate just before using the MP TROLL 9500. However, if the instrument will not be put to use immediately, store it as follows:

- Leave the sensors installed. Remove the Cal Cup and rinse it and the sensors. Add about 50-100 mL of tap water to the Cal Cup. Return the probe to the Cal Cup for transport to the field.

**CALIBRATE THE EXTENDED SENSORS**

For some sensors, the Quick-Cal procedure is not available. A traditional calibration is recommended. To calibrate, select the sensor in the Parameters list and click or tap Calibrate to launch a Calibration Wizard. Complete calibration information is in the following sections of this manual:

<table>
<thead>
<tr>
<th>Sensor</th>
<th>Section</th>
</tr>
</thead>
<tbody>
<tr>
<td>RDO Optical Dissolved Oxygen</td>
<td>13 (second part)</td>
</tr>
<tr>
<td>Ammonium</td>
<td>15</td>
</tr>
<tr>
<td>Chloride</td>
<td>16</td>
</tr>
<tr>
<td>Nitrate</td>
<td>17</td>
</tr>
<tr>
<td>Turbidity</td>
<td>18</td>
</tr>
</tbody>
</table>

**SET UP CUSTOM PRESSURE/LEVEL MEASUREMENTS**

You can configure a pressure channel to display measurements just the way you want them—as raw pressure head above the sensor, as depth, or as water level with a reference. The specified settings are easily changed from one mode to another, and any choice can be redone or undone later, when viewing test data.

1. With the MP TROLL 9500 connected to a host PC, select the pressure parameter in the Navigation tree.
2. Click or tap Edit....

The Parameter Wizard will help you enter the required information. The choices are explained in greater detail in Section 7 below.

3. When you finish the Wizard, the information is written to the device and the display is updated. The pressure channel is ready to take measurements.

**TIP:** For complete information on setting up the pressure parameter, refer to Section 7 of this operator's manual.
SET UP TO LOG DATA (SET UP A TEST)

A “test” is a set of instructions to the instrument’s internal data logger for collecting a set of data—including which parameters to measure, how often, and when to start. Section 6 below contains detailed information on setting up tests.

1. With the MP TROLL 9500 connected to a host PC, click or tap Tests in the Navigation tree.

2. Click or tap Add....

The Test Wizard will help you enter the information. The choices are discussed in detail in Section 6 below.

3. When you finish the Wizard, Win-Situ sends the information to the device and updates the display.

   If you added a “scheduled” test (indicated by in the Navigation tree), it will start at its programmed time.

   If you set up a test or a manual start (indicated by in the Navigation tree), you can start it at any time by pressing the Start button while connected in software.

   TIP: For complete information on setting up tests, refer to Section 7 of this operator’s manual.

COMPLETE THE SETUP

If real-time readings are not required, remove the TROLL Com from the cable after programming.

Protect the “uphole” end of the cable with the cable dust cap or desiccant.

The cable dust cap is not waterproof. Be sure it is positioned above the highest anticipated water level. Avoid areas that may flood.

If cable will not be used, attach a Twist-Lock Hanger to the TROLL 9500.

The instrument is now ready for transport to the site where it will be used to take measurements.
The interface from a desktop or laptop computer (PC) to the Multi-Parameter TROLL 9500 is provided by instrument control software called Win-Situ® 4. Instrument control is accomplished through a familiar, easy-to-use Navigation Tree interface.

For field use on a hand-held computer (PDA), optional Pocket-Situ 4 provides all of Win-Situ’s functionality and features in a convenient field-portable platform.

Use Win-Situ or Pocket-Situ for these tasks:

- to calibrate water-quality sensors
- to convert pressure measurements to depth or level readings
- to set up data collection schedules (“tests”)
- to take direct “manual” readings
- to take continuous real-time readings (Profiling)
- to transfer data from the Multi-Parameter TROLL 9500 to the host computer
- to view logged data in text or graph format
- to delete data from the Multi-Parameter TROLL 9500 in order to free up memory
- to monitor battery and memory usage
- to set the device clock
- to upgrade device firmware (when available)
- to choose measurement units and other custom display options
- to monitor indicator water-quality parameters during low-flow pumping
- to schedule automatic remote transmissions (if available)

TIP: For software system requirements, see Section 2, Components & Features. Software installation is covered in Section 3, Getting Started.

LAUNCHING THE SOFTWARE

WIN-SITU 4

Use one of the following methods:

- Double-click the shortcut created on the desktop during installation.
- Select Win-Situ 4 from the In-Situ Inc program group on the Windows Start menu.

POCKET-SITU 4

Tap Start to display the RuggedReader’s Start Menu. Select Pocket-Situ 4.

- If you don’t see a shortcut, tap Programs and select Pocket-Situ 4
- If it launches in “demo” mode, you can activate it with the license key and activation code from your In-Situ License Agreement
THE USER INTERFACE

Win-Situ 4 and Pocket-Situ 4 use the familiar “Navigation tree” to display your instrument network. The tree appears on the left of the screen in Win-Situ, and at the top of the screen in Pocket-Situ. The remainder of the screen is an “information pane” with details about the “node” you have selected in the tree. The selected node and its information constitute a “view” in the interface.

THE NAVIGATION TREE

At the top of the tree is the “Home” site (the host computer). Below this is the Flow-Sense Wizard (see Section 20), then the Data Folder containing tests that have been extracted from the device to the host computer. This is followed by one or more connections (COM ports), and then one or more devices (Multi-Parameter TROLLs or other In-Situ instruments) accessible through each COM port. The Device node is further expandable to show the parameters the device can measure and the tests contained in the device’s memory.

The site, wizard, data folder, connection, devices, parameters, and tests are all “nodes” in the Navigation tree. The symbol + means a node can be expanded to show more nodes below it. Each selection results in a unique “view” in the Navigation tree.
SOFTWARE FUNCTIONS

CONFIGURING THE PORT

When you start Win-Situ or Pocket-Situ for the first time, a new, empty site called “Home” is displayed at the top of the Navigation tree. The Connection Wizard starts automatically to help you add a connection to the site, specifying your computer’s COM port, desired baud rate, and other port properties.

To add a new connection to the site—or if the Connection Wizard does not start automatically for some reason—proceed as follows:

1. Select the site.
2. Click or tap Add....

To change the properties of a connection:

1. Select the connection.
2. Click or tap Edit....

To delete a connection from the site:

1. Select the connection.
2. Click or tap Delete.

For additional information on the port properties prompted by the Connection Wizard, refer to Section 3, Getting Started.

ESTABLISHING COMMUNICATION WITH THE MP TROLL 9500

New Connection

The last option in the Connection Wizard is to Connect and find devices on “Finish.” If you select this option, the software will automatically establish communication with the MP TROLL 9500.

If you did not select this option, proceed as for an existing connection.

Existing Connection

Each time you launch the software, the site and the connection saved in your last session are displayed in the Navigation tree. To connect:

1. Select the connection.
2. Click Find. Or just double-click the connection.

Win-Situ or Pocket-Situ will open the connection, synchronize the baud rate, locate the device, and display it in the Navigation tree.

3. Click to select the MP TROLL 9500 in the Navigation tree.

   If the device’s remaining battery capacity is 5% or less, the device may not be displayed in the tree. Replace the batteries before continuing.

DISPLAYING DEVICE INFORMATION

When you select the MP TROLL 9500 in the Navigation tree, the software first retrieves information about the device, then automatically detects the installed sensors and displays them in the tree.

TIP: If one or more sensors is incorrectly installed, an error message will be displayed. Simply remove the sensor and install it in the correct position, then “refresh” the device to display the sensors in the Parameters list.

With the MP TROLL selected in the Navigation tree, the Information pane displays a wealth of information about the device, as shown in the Win-Situ screen on the following page. You may need to scroll to view all the information in Pocket-Situ.
The Information pane also provides buttons for actions you can perform, such as upgrading the device features (if available), editing the device properties (including the real-time clock), and refreshing the connection.

Battery Capacity display: On battery power, the "loaded" battery voltage is reported (i.e., the device is drawing current from the batteries).

If external power is detected, an artificially high battery voltage may be reported depending on the device hardware. Hardware versions earlier than 3 report a constant, regulated voltage (approximately 3.3 V). Hardware version 3 will report the true battery voltage, but since switching to external power removes the load from the battery the number may change when you switch from external to battery and vice-versa.

UPGRADING FIRMWARE AND FEATURES

Firmware Upgrade

New software releases may include a new version of firmware for the MP TROLL 9500. If the software detects a newer version of firmware than that currently loaded in the device, you will be prompted to upgrade device firmware when you connect to the MP TROLL 9500. The upgrade process is brief and software-assisted.

For best performance, we recommend you upgrade to new firmware when prompted by the software.

TIP: In order for the software to detect it, the new firmware must be located in the Firmware folder in the folder where Win-Situ or Pocket-Situ is installed. Firmware is automatically copied to these locations when new software is installed.

Features Upgrade

The MP TROLL 9500 is available in several models, each offering a different feature set. Some models may be field-upgraded to take advantage of a wider range of features. The field upgrade involves keying in an upgrade code, issued by In-Situ when an upgrade is purchased for a specific instrument serial number.

If you have an upgradable feature set, the Upgrade button will be available in the Information pane when the device is selected in the Navigation tree. If you have purchased a features upgrade for your MP TROLL 9500, press Upgrade and key in the upgrade code issued by In-Situ Inc. Follow the instructions in the Upgrade Wizard.

Device View of an MP TROLL 9500

The device displays its "family," TROLL 9000, in the Navigation tree.
EDITING THE DEVICE PROPERTIES

Setting a Device Name

The software recognizes each device on the network by its type and serial number. In addition, you can assign a meaningful description—a well or site name, location coordinates, etc. This name will be displayed in the Navigation tree and in the header of test data files.

1. With the MP TROLL 9500 connected to a host PC and selected in the Navigation tree, press Edit...
2. In the Device Wizard, select Name, then Next.
3. Enter a new name for the device (up to 16 characters).
4. When you finish the Wizard, the information is sent to the device and the display is updated.

Setting the Real-Time Clock

Data collection schedules depend on the device clock, shown in the lower right corner of the desktop PC interface. If the clock is not correct, set it as follows:

1. With the MP TROLL 9500 connected to a host PC and selected in the Navigation tree, press Edit....
2. In the Device Wizard, select Clock, then Next.
3. Follow the instructions to synchronize the MP TROLL 9500’s internal real-time clock to the host computer.
4. When you finish the Wizard, the information is sent to the device and the display is updated.

Setting Battery Information

After replacing the batteries, update the device battery information:

1. With the MP TROLL 9500 connected to a host PC and selected in the Navigation tree, press Edit....
2. In the Device Wizard, select Battery Information, then Next.
3. Select the appropriate battery type and enter the installation date.
4. When you finish the Wizard, the information is sent to the device and the display is updated.

SDI-12 and ASCII Mode Preferences

Preferences for these communication modes may be set in the Device Wizard.

1. With the MP TROLL 9500 connected to a host PC and selected in the Navigation tree, press Edit....
2. In the Device Wizard, select the desired mode to edit, then Next.
3. Change the settings as appropriate.

To communicate via SDI-12, insure SDI-12 is enabled (factory default) in the device. For telemetry applications, SDI-12 should be disabled.

EDITING THE DEVICE PROPERTIES

1. Select the device in the Navigation tree and click Edit.
2. Select the device property to edit.
CHOOSING MEASUREMENT UNITS AND OTHER PREFERENCES

Preferences can be set at any time, and are saved with the site. A connection to the instrument is not required.

User-selectable preferences include:

- Parameter measurement units & elapsed time units
- Data file view—report or graph
- Calendar date & time of day format
- Metric or English units for the Flow-Sense Wizard

The following Settings Options are also available:

- Start the application in Profiler mode
- Specify desired Profiling rate
- Re-display the “Don’t Ask Again” dialogs

To display the Preferences window, do one of the following:

- In Win-Situ, select Preferences from the Options menu.
- In Pocket-Situ:
  a. Tap the Home site in the Navigation tree...
  b. then tap Setup... in the command bar.

Units

Default units are used for the display of data unless you specify other preferences. To set unit preferences, first display the Preferences window as above.

1. Select the Units tab.
2. In the top of the Units window, select a parameter whose unit you wish to change.
3. In the bottom of the window, select the unit you want for the highlighted measurement. Repeat for as many units as you like.
4. Do one of the following:
   - Click OK to apply the new units and close the window.
   - Click the Settings tab to change other settings.

Other Preferences

To change other aspects of Win-Situ/Pocket-Situ operation, first display the Preferences window as above.

1. Click the Settings tab.
2. Select the options you want:
   - Start application in Profiler mode. This will take effect at your next session.
   - Expand Devices on Find. This will take effect the next time you click “Find.” The software will automatically display all nodes in the device tree without any further action from you.
   - Set desired Profiling rate—the Profiler default rate is 2 seconds, but you may select any rate from 2 to 60 seconds.
   - Re-display the “Don’t Ask Again” dialogs. This appears if you have selected “Don’t ask me this again” in any Win-Situ or Pocket-Situ dialog boxes.

Boxcar filtering is no longer available.

3. Select OK to close the window and apply the settings.

TIP: Preferences are saved with the site (on the PC), not in the device.
CHOOSING PRESSURE DISPLAY & CONVERSION OPTIONS

Measurements from the optional pressure sensor may be displayed as raw pressure head above the sensor, as depth, or as water level with a reference. When measuring depth or level, you have a choice of methods for conversion from pressure measurements. The specified settings are easily changed from one mode to another, and most choices can be redone or undone later, when viewing test data.

TIP: For more on the pressure channel setup, refer to Section 7.

To set the custom display options and pressure-to-level conversion options for a pressure channel:

1. With the MP TROLL 9500 connected to a host PC and selected in the Navigation tree, expand the Parameters node and select the pressure parameter.

2. Click Edit....

The Parameter Wizard starts. Refer to Section 7 for complete information on the display and conversion options.

3. When you finish the Wizard, the information is sent to the device and the display is updated.

PERFORMING WATER-QUALITY CALIBRATIONS

Although satisfactory results may be achieved in some cases without field-calibrating the water-quality sensors, for best results we recommend a preliminary calibration procedure before the first field use, and periodic checks and recalibrations as necessary.

The software provides several options for sensor calibration. Choose a method based on the time you are willing to spend calibrating and the accuracy you wish to achieve.

- **Traditional Calibration** guided by software wizards can achieve accuracy and resolution equivalent to laboratory-based meters. Some sensors require a single-point calibration, some present a choice of single- or multi-point, requiring more than one calibration standard.

  A detailed, step-by-step description of each water quality calibration may be found in the relevant parameter section (Sections 11-18) of this manual.

- **Quick Cal** calibrates the basic sensors (pH, ORP, polarographic D.O., conductivity) simultaneously to achieve adequate performance with minimal labor.

  Refer to Section 3, Getting Started, for the Quick Cal procedure.
LOGGING DATA (ADDING A TEST)

Logging data with the MP TROLL 9500 is called “running a test.” To tell the instrument how to run the test—which parameters to measure, how often, when to start, etc.—you “add” a test to the device.

**Tip:** For more on logging, extracting, viewing, and deleting data, refer to Section 6.

1. With the MP TROLL 9500 connected to a host PC and selected in the Navigation tree, click or tap Tests.
2. Click or tap Add.

![Test Wizard](image)

The Test Wizard starts. See Section 6 for a description of the elements that constitute a complete test definition.

3. When you finish the Wizard, the information is sent to the device and the display is updated.

TAKING “MANUAL” READINGS

The MP TROLL 9500 collects data in “tests” but you can get a quick “manual” reading from any device apart from a test, even while a test is running.

1. With the MP TROLL 9500 connected to a host PC and selected in the Navigation tree, click or tap Parameters.
2. In the Information pane, select one or more parameters to read. To select multiple parameters, hold the Control key while selecting. (To display the CTRL key on a PDA, tap the keyboard symbol in the Command bar.)

![Parameter Information](image)

3. Click or tap Read.

**Tip:** For continuously updated real-time “manual” readings of all parameters, select Profiler in the Information pane. Refer to Section 5 for Profiler information.
EXTRACTING AND VIEWING DATA

Logged data ("tests") reside in the MP TROLL 9500 until you “extract” them. The extract operation copies test data from the device memory to the host computer. Once a test has been extracted to the host computer, it can be viewed. A test can be extracted and viewed at any time, even while it or another test is running.

**TIP:** For more on logging, extracting, viewing, and deleting data, refer to Section 6.

To extract a test:

1. With the MP TROLL 9500 connected to a host PC and selected in the Navigation tree, expand the Tests node to display all the tests in the device.
2. Select a test and click or tap *Extract*.
3. To view the test immediately, select the *View* option at the end of the download.

**TIP:** After the test has been extracted to the host computer, it will appear in the Data Folder node in the Navigation tree. A connection to the instrument is not required to view the extracted data.

DELETING DATA FROM INSTRUMENT MEMORY

The Extract operation copies a test data file to the host PC, but does not remove it from the MP TROLL 9500. Test data files remain in the Multi-Parameter TROLL 9500’s memory until you delete them. Deleting tests frees up device memory.

**CAUTION:** Be sure to extract data you want to save before deleting from the device. Once a test is deleted, the data cannot be retrieved!

1. With the MP TROLL 9500 connected to a host PC and selected in the Navigation tree, expand the Tests node to display all the tests in the device.
2. Select a test and click *Delete*.
3. Click *Yes* to confirm your selection.

The selected test is deleted and the display is updated.

**TIP:** If a test is running in the device, the Delete button is not available. You must stop the running test before tests can be deleted. See “Stopping a Test” in Section 6.

EXITING THE SOFTWARE

WIN-SITU 4

Select Exit from the File menu.

POCKET-SITU 4

1. Tap the Home site in the Navigation tree...
2. then tap Exit in the command bar.

**CAUTION:** Be sure to Exit Pocket-Situ as described above after each session. This releases allocated resources used by Pocket-Situ, assuring that the COM port is available the next time you connect to the MP TROLL 9500, or to a desktop PC using ActiveSync.
All models of the Multi-Parameter TROLL 9500 include a software feature called the Profiler that delivers instant real-time readings on all available channels. Readings are continuously updated as long as the Profiler is running. This provides a convenient way to characterize surface waters using a hand-held controller running Pocket-Situ.

**PROFILER FEATURES**

- Auto detect—the software automatically identifies all properly connected sensors
- Continuously updated readings from all available channels
- Optional data logging to the connected PC
- Optional start device in Profiler mode
- Optional selectable Profiler interval (2-60 seconds)

You will need—

- MP TROLL 9500 with water quality sensors installed and calibrated
- RuggedCable
- Cable connect TROLL Com (RS232 for RuggedReader)
- Laptop PC running Win-Situ 4 or RuggedReader running Pocket-Situ 4

**STARTING THE PROFILER**

1. Connect the MP TROLL 9500 to a host PC and establish communication in Win-Situ 4 or Pocket-Situ 4.

2. Click to select the MP TROLL 9500 in the Navigation tree. The software will automatically detect the installed sensors and display them in the Information pane.

   **TIP:** If one or more sensors is incorrectly installed, an error message will be displayed. Remove the sensor, install it in the correct position, and refresh the display before continuing.

3. Click or tap Parameters in the Navigation tree.

4. Click or tap **Profiler** to start the Profiler.

5. You will be asked if you want to set a filename for logged Profiler data (logging the data is optional)

   - To use the default name click **OK**.
   - To specify another name, key it in and click **OK**.
   - To avoid this question each time you start the Profiler, check the option box and click **OK**.

   In a moment, each active channel will be read sequentially, and the readings will be displayed. Up to 8 channels can be shown.

**TIP:** Don’t let the PDA time out while Profiling. To locate this setting in most PDAs, display the Start menu, select Settings, System tab, Power.
Readings are updated approximately every 2 seconds as the Profiler cycles through the available channels in turn.

The currently selected measurement unit is shown below each reading.

**TIP:** You can use the Setup option to change the Profiler’s reading rate and/or the displayed units. See Customizing the Profiler later in this section for details.

A window without a reading indicates the channel is not available to read, usually because there is no sensor installed in the port.

**TIP:** If a channel you wish to see is not displayed, click the symbol below a reading you can do without and select the channel you want to add from the list of available channels. This is particularly useful for RDO readings.

**GRAPHING PROFILER DATA**

To see a graph of any channel during the current Profiler session, do one of the following:

- Click or tap any reading.
- Click or tap **Graph**, and select the channel from the drop-down list.

Readings for the selected channel since the start of profiling will be displayed in graphical format.

The profiler can graph up to 300 readings. A range slider (scroll bar) on the right side of the graph allows you to change the range of visible data. Like the channel display, the graph will be continuously updated. A range slider at the bottom of the graph lets you zoom in on a narrower time period.

To switch back to the display of active channels, click **Meter**.

**PROFILING DISSOLVED OXYGEN**

The ability of the polarographic D.O. sensor to respond to a change in the dissolved oxygen content of the medium—for the sensor to accurately “see” a change—varies with the membrane thickness.

- A 1-mil membrane responds in about 1–2 minutes to significant DO changes;
- A 2-mil membrane responds in about 1.5–3 minutes to significant DO changes.

The RDO Optical D.O. sensor has a response time of $T(90) = 30$ sec and $T(95) = 37$.

**PROFILING PRESSURE OR DEPTH**

Profiler readings from a pressure channel are displayed as pressure head or depth according to the pressure channel setup. Refer to Section 7 below for pressure display and conversion options. Level mode readings with a user-entered reference are primarily for use in aquifer tests.

**TIP:** Profiling with the MP TROLL’s default pressure settings (no channel setup) will display pressure head in psi (pounds per square inch). To display depth, edit the pressure channel as described in Section 7.

**PROFILING TURBIDITY**

The turbidity sensor requires a 5-second warmup before the first reading. Subsequent readings do not require a warmup.

If a turbidity wiper accessory is installed in the TROLL 9500, it performs an initial wipe of the sensor optics—this takes about 15 seconds—then displays the first turbidity reading. If the profiling rate is longer than 15 seconds, this 15 second wipe will happen before each reading. To avoid this delay, set the profiling rate to less than 15 seconds. See Customizing the Profiler later in this section for details.

Section 18 below has additional information on the turbidity wiper.
SECTION 5: PROFILING

Display a graph of the data, starting with the first channel.

Log next set of readings to the Data Folder on the PC.

Select a different channel for display in this position.

Click any reading to display a graph of the channel.

Log continuously updated readings to the Data Folder on the PC. Changes to “Stop Log” during continuous logging.

Close the Profiler and return to the Parameter display.

Profiler logging status

Display a graph of the data, starting with the first channel.

Up to 300 readings (including the current reading) can be graphed.

X axis range slider

Auto-scales the Y axis for the selected range (available for some channels).

Y axis range slider.

Double-click to zoom fully out; double-click again to restore.

Select the channel to graph.

Return to the Profiler's channel display.

Close the Profiler and return to the Parameter view.
CUSTOMIZING THE PROFILER

Changing the Channels Displayed

The arrow < below each Profiler reading displays a drop-down list that may be used to assign a different channel to each position, if desired. You can use this button to add a channel, such as RDO Optical Dissolved Oxygen, that is not displayed by default. However, only 8 channels can be shown.

Changing Measurement Units

Close the Profiler. In Win-Situ, select Preferences on the Options menu. In Pocket-Situ, select the Home site, then tap Setup in the command bar.

Changing the Sample Rate

Close the Profiler. In Win-Situ, select Preferences on the Options menu, then select the Settings tab. In Pocket-Situ, select the Home site, tap Setup in the command bar, then select the Settings tab. You may select any sample rate between 2 and 60 seconds.

Starting in Profiler Mode

In Win-Situ, select Preferences on the Options menu, then select the Settings tab. In Pocket-Situ, select the Home site, tap Setup in the command bar, then select the Settings tab. Check ✔ Start application in Profiler mode. This will take effect at your next session (or exit and re-start to apply this setting).

LOGGING PROFILER DATA

Profiler data may be logged to the connected PC while in the “Meter” view.

- **Snapshot**: To log a single set of Profiler readings, click or tap the SnapShot button in the “Meter” view.

- **Continuous**: To log continuous readings, click or tap the Continuous button in the “Meter” view. Readings will be logged until you cancel the operation by clicking Stop Log.

TO STOP LOGGING

The SnapShot function logs a single set of readings and stops logging automatically.

In Continuous mode, click Stop Log. Note that this does not stop Profiling; readings will continue to be updated on the screen.

RETRIEVING LOGGED PROFILER DATA

In either Snapshot or Continuous mode, Profiler data are logged to the connected PC and accessible through the Data Folder. The data may be retrieved at any time. A connection to the instrument is not necessary.

To retrieve logged Profiler data:

1. Expand the Data Folder in the Navigation tree by tapping the +.
2. Expand the node for the device type and serial number.
3. Look for a data file named with date and time of the profiler reading, and with the ending profiler.bin—or whatever custom name you may have specified when starting the Profiler.

TIP: The time shown in the filename of a Profiler log is the time the Profiler started, which may be different from the time of the first data point logged. Similarly, the Elapsed Time indicated in the Report view of a continuous Profiler log is the elapsed time since the Profiler started.

For additional information on viewing data in the Data Folder, see the section “Viewing Logged Data” at the end of Section 6 below.

EXITING THE PROFILER

When you are ready to exit the Profiler, click Close. The Parameters view will return to the screen.
6 LOGGING DATA

Memory-equipped Professional models of the MP TROLL 9500 are ideal for monitoring and recording water quality data. Logging schedules can be set up in advance, and the collected data stored in the device’s storage memory until needed.

Collecting a set of data with the instrument is called “running a test.” A “test” is initially a set of user-defined instructions to the logger about how to collect the data:

- which parameters to measure
- how often to measure
- when to start taking and logging measurements

After it has “run,” the completed test consists of a data file that was logged following the instructions above.

Many of the things you do through Win-Situ or Pocket-Situ control software can be visualized as actions performed on tests. For example,

- to give the device instructions for collecting data: add a test.
- when you want the device to end data collection: stop a test.
- to copy test data from the device to a host computer: extract the test.

These actions are initiated by buttons in the Information pane when the device’s Tests node (group view of all tests) or a Single test is selected in the Navigation tree.

**Advantages of the Tests node (group view):** (1) the Add action is available, and (2) group operations such as delete and extract can be performed on multiple tests simultaneously. For illustrations, see the box on page 39.

**ADDING A TEST TO A DEVICE: THE TEST WIZARD**

The Add action programs the MP TROLL to collect data. The Test Wizard starts automatically to help you enter the required information.

1. With the MP TROLL 9500 connected to a host PC and selected in the Navigation tree, click or tap Tests.
2. Click or tap Add. The Test Wizard starts. The choices are discussed below.
3. When you finish the Wizard, the information is written to the device and the display is updated.

---

**TIP:** Defining a test, setting up a test, adding a test—these terms all mean the same thing and may be used interchangeably in this manual.
With a D.O. sensor installed (polarographic or optical)—

Before the Test Wizard starts, you will be asked how you want to handle barometric pressure. Barometric pressure is important in converting measurement of D.O. concentration to percent saturation. If the TROLL 9500 cable is vented, an accurate barometric pressure value can be read from the onboard barometric pressure sensor. If the TROLL cable is non-vented, then a barometric pressure value should be entered manually.

The dialog box shown below asks if you wish to edit the barometric settings in the absence of vented cable. Do one of the following:

• If the TROLL 9500 is on vented cable now and will take measurements using vented cable, click No — and you may want to check the “Don’t ask me this again” box.
• If the device is on vented cable now but will take measurements using non-vented cable, click Yes. In the Edit Barometric Channel screen, check the box indicating non-vented cable for measurements but vented cable for calibration/programming.
• If the device is on non-vented cable now and will take measurements on non-vented cable, click Yes. In the Edit Barometric Channel screen, check the box indicating non-vented cable for measurements and enter a barometric pressure value. For help in supplying information if the cable is not vented, see Section 9, Monitoring Barometric Pressure.

TEST SETUP PARAMETERS

A complete test definition has five parts. The choices available in each screen are tailored to reflect your selection in earlier screens.

1. Test Name (Optional)

You may enter up to 16 characters to identify the test. A default name is proposed. The test name is displayed in the Navigation tree, in the test data file, and in the filename of tests extracted from the device to the Data Folder on the host PC.

2. Parameters to Include

All available parameters are selected by default. De-select any parameter you do not want to measure during this test by clearing the checkbox. Remaining choices will be tailored to your selection.

The barometric pressure and temperature channels are automatically included when water-quality channels are selected so that their readings are available for compensating the measurements.

With an RDO® optical D.O. sensor installed—

Before the Test Wizard starts, you will be asked how you want to handle salinity compensation. The sensor does not react to changes in salinity. To compensate for the lower solubility of oxygen in salty water, a salinity value stored in the sensor can be applied. This ensures that the reported concentration of dissolved oxygen in the presence of significant water salinity is accurate. The compensation algorithm is applied internally before concentration is reported by the sensor. Choose one of the following:

• Retain Current Setting (shown below).
• Use Current Conductivity Reading: If a conductivity sensor is present, you may select the second option. However, the conductivity sensor is subject to fouling, and any conductivity errors thus created will be transmitted to the RDO sensor.
• User Input Value: When working in waters with a constant and known salinity, and a conductivity sensor is not present, select this option and enter the salinity value (in PSU, Practical Salinity Units). The fixed value is effective in situations where the salinity variability is low. The value can be changed at any time by selecting the RDO sensor in the Navigation tree and clicking “Edit,” then Edit Salinity.

3. Measurement Schedule

The available schedules depend on your device’s firmware and the parameters selected for inclusion in the test. This list is a quick overview. Additional details are given later in this section.

• Linear. All measurements are evenly spaced at a user-specified interval, and all measurements are stored in the device memory.
• Event. Measurements are evenly spaced, but you can record an “event” and conserve memory by having the device store only measurements that differ from the previous stored measurement of a designated parameter by a specified amount. Any available
parameter may be selected for event comparisons, and the “delta” or difference amount is prompted in the current basic parameter unit.

- **Linear Average.** This type of test can smooth out anomalous highs and lows that may occur, for example, when a water wave passes over the instrument. Each stored measurement is the average of several rapid measurements.

### 4. Measurement Interval

The interval specifies how often measurements will be taken during this test. Enter any combination of Days, Hours, Minutes, & Seconds. The minimum (fastest) allowable interval depends on your choice of measurement schedule and the parameters included in the test. For most parameters, the minimum interval is 5 seconds. If RDO Optical Dissolved Oxygen is included, the minimum interval is 10 seconds.

### 5. Start Mode

Choose one of the following options for starting this test:

- **Manual.** A manual test can be started at any time while the MP TROLL 9500 is connected to a PC by pressing the **Start** button. This mode is useful when you want to synchronize the start of data collection with an external event like starting a pump.

- **Scheduled.** A scheduled start test will start at the date and time you specify. The time proposed by the software is the next hour on the hour, calculated from the current device time plus 10 minutes. Arrows beside the list boxes may be used to change the start time and date, or key in the desired start time and date.

**TIP:** In entering the Scheduled Start time, keep in mind that when this test starts, it will stop a running test.

The time proposed for a Scheduled start is the next hour on the hour, calculated from the current device time plus 10 minutes. In Pocket-Situ this is your only indication of the device time. If the proposed time is wrong, cancel test setup and set the device clock before scheduling tests.

### ENDING SETUP

After you select the test options and click **Finish**, the information will be sent to the device and the new test will appear in the Navigation tree.

To let you know which tests can be started with the Start button, the software displays a different symbol for each start type:

- Manual start test (you start)
- Scheduled start test (starts by itself)

### More on Measurement Schedules

**Linear**

In Linear sampling, all selected channels are measured at the same Measurement Interval; all measurements are stored in memory.

**Event**

All selected parameters are measured at your chosen Measurement Interval, but a data point (reading from all active channels) is stored only if the measurement on the designated “event” channel exceeds a user-defined value. This value is called “Delta” because it relates to a change in the measurement. Here’s how it works:

**Delta**

Each measurement on the selected “event” channel (pressure, for example) is compared to a reference. The initial reference is taken at the start of the test. When a pressure measurement varies from the reference by less than the Delta amount, the data point is not stored. When a pressure measurement varies from the reference by more than the Delta amount, the data point (all channel measurements) is stored. The stored pressure measurement is called a “Delta point” and becomes the new reference for comparison.

In an Event test, then, small and essentially insignificant changes are not stored, but larger and more significant changes are. This scheme will minimize the size of a data file by storing meaningful data.
EVENT (continued)

Minimum Delta Values

System accuracy is a function of many variables. It is theoretically possible to choose a Delta value so small that random system noise, however slight, might “look” significant. We recommend that you enter a reasonable Delta value for the parameter being measured to avoid triggering an event storage when no event has occurred.

TIP: Units for the Event channel should be set to the “basic” parameter unit (e.g., psi). Derived (software-calculated) units are not available.

Default Storage

Consider a situation where there is virtually no change through time in measured value. In this case, it is possible that almost no data would be stored to the data file. To avoid the possibility that a test may contain little or no stored data, sparse linear sampling also occurs whereby data points are stored to the data file every \( n \) measurements regardless of the measurement comparisons that are occurring. This type of data is called “default storage.” For example, if the Measurement Interval is 10 minutes and you specify a Default storage every 6 measurements—as shown in the event test setup screen below—then data will be logged every hour regardless of the Delta comparisons.

Data gathered in an Event test consist of both “Delta points” and “default storage.” There is no distinction made between them in the actual data file since they both represent measurements of the same physical property (pressure, temperature, conductivity, etc.).

**Linear Average**

This type of test can eliminate anomalous highs and lows that may occur—for example, if a wave passes over the instrument at the time a measurement is made. For every measurement stored in the data file, the device takes a specified number of measurements at a specified interval, averages them, and stores the average value.

You are prompted for:

- **A** the storage interval (how often an averaged measurement will be stored in the data file)
- **B** the interval between the rapid measurements that will be averaged (1-255 seconds)
- **C** the number of measurements that will be averaged for each stored measurement (2-99)

TIP: The storage interval (A) must be at least twice as long as the rapid interval (B) times the number of measurements for averaging (C). In the example shown below, 5 seconds \( \times 3 = 15 \) seconds, so the storage interval would have to be at least 30 seconds.

In the linear average test example shown in the screen below, a measurement will be logged every hour (A). The logged measurement will consist of the average of 3 measurements (C) taken 5 seconds apart (B).

---

**Linear Average Test Setup**

![Linear Average Test Setup](image-url)
OTHER TEST OPERATIONS

There are six basic test operations:

- **Add**—programs the instrument to collect data. See the first part of this section for details.
- **Edit**—allows you to change the pre-programmed test setup before the test runs. Editing a test launches the Test Wizard. See the first part of this section for details.
- **Start**—starts the selected test if it has been defined for a manual start.
- **Stop**—stops a running test.
- **Extract**—copies the selected test data from the instrument to the host computer. A copy of the data remains in the instrument.
- **Delete**—deletes the selected test from the instrument.
- **Clone**—makes a copy of the selected test definition setup.

The operations available depend on the status of the selected test. A different symbol is displayed for each test status:

- **Pending test**—manual start
- **Completed test**
- **Running test**
- **Pending test**—scheduled start
- **ABEND test**

(came to an ABnormal END, e.g., by losing power or filling the memory)
STARTING A MANUAL START TEST

1. With the MP TROLL 9500 connected to a host C, select a pending manual test in the Navigation tree.

2. Click or tap Start.

   TIP: If a test is running in the device, the Start button is not available. You must stop the running test before you can start a new one. See “Stopping a Test” below.

3. You will be asked to confirm your selection.

4. After you confirm, the test starts and the display is updated.

   TIP: Once a test starts, the following actions will not be available: Add, Delete, Edit (device, parameter, test), Clone, Calibrate.

STOPPING A TEST

The end of a test cannot be programmed in advance. In most cases, the Stop button is the only way to stop a running test. Exceptions: A test stops automatically when any of the following occurs:

- when another test starts
- if the device memory fills up
- if power is lost.

1. With the MP TROLL 9500 connected to a host PC, select a running test in the Navigation tree.

2. Click or tap Stop. You will be asked to confirm your selection.

3. After you confirm, the test stops and the display is updated.

RETREIVING LOGGED DATA

Retrieving, downloading, uploading, transferring—these terms are sometimes used interchangeably to mean the act of copying data from the place where it was logged to a host PC. The Extract operation in Win-Situ or Pocket-Situ retrieves test data from the MP TROLL 9500 memory and saves it to a file on the host computer. You may view the file immediately after the download, or later.

1. With the MP TROLL 9500 connected to a host PC and selected in the Navigation tree, click or tap Tests.

2. In the Information pane, select one or more tests to extract. To select multiple tests, hold the CTRL key while selecting. (To display the CTRL key on a PDA, tap the keyboard symbol.)

3. Click or tap Extract to extract the test(s).

   When the extraction is complete, the name(s) and locations of the test data file(s) on the host computer are displayed (see box on the next page). If a selected test has been extracted previously, only data logged since the last download is extracted and is automatically appended to the original file.

   The software presents two options after the extraction:

   - View launches the data viewer to view the selected file.
   - Done returns to the Tests view in the software.

   TIP: The “Extract” operation puts a copy of a test data file on your PC, but does not delete the test from the device. To free up device memory, see Deleting Tests, next.

   TIP: When extracting a large test to a PDA on battery power, avoid letting the PDA time out during the transfer. To locate this setting in most PDAs, display the Start menu, select Settings, System tab, Power.

Q: Where do my extracted data files go?

Win-Situ 4 extracts tests to a folder named “Data” in the folder where Win-Situ 4 is installed. The file structure looks like this:

```
Win-Situ \ Data \ SN30701  2004-02-12  150000  Test #3.bin
```

<table>
<thead>
<tr>
<th>Device serial number</th>
<th>Test start date (yyyy-mm-dd)</th>
<th>Test start time (hhmmss)</th>
<th>Test name</th>
<th>File type</th>
</tr>
</thead>
</table>

TIP: On a PDA, the Pocket-Situ \ Data folder is located in the device’s non-volatile storage.
DELETING TESTS
Tests are stored in the device memory until you delete them. The Delete operation permanently removes selected tests from the device, and reorganizes the memory to optimize future data storage. Depending on how full the memory is, the process may take several minutes.

1. With the MP TROLL 9500 connected to a host PC and selected in the Navigation tree, click or tap Tests.
2. In the Information pane, select one or more tests to delete. To select multiple tests, hold the CTRL key while selecting. (To display the CTRL key on a PDA, tap the keyboard symbol in the Command bar.)
3. Click or tap Delete. You will be asked to confirm your selection.

**TIP:** If a test is running in the device, the Delete button is not available. You must stop the running test before tests can be deleted. See “Stopping a Test” above.

4. After you confirm, the selected test(s) are permanently removed from the device.

**TIP:** The Delete procedure can be used to cancel a pending scheduled test.

CLONING A TEST
The Clone function lets you copy all the elements of an existing test—the test name, selected channels, measurement schedule and interval—without having to define a new test “from scratch.” You can clone a pending test or a completed test.

1. With the MP TROLL 9500 connected to a host PC and selected in the Navigation tree, click or tap Tests.
2. In the Information pane, select a test to clone.

3. Click or tap Clone to preview the test definition. The Wizard will still present all the screens, but you can just click Next to get through them quickly (or make any changes you want).
4. Click Finish to end the wizard. The information will be sent to the device and the new test will appear in the Navigation tree.

**TIP:** The proposed start type for a cloned test will always be Manual, regardless of the start type of the original test. This avoids scheduling a test to start in the past.
**TRANSFERRING FILES FROM A PDA TO A DESKTOP PC**

If Win-Situ Sync (or its earlier release, Pocket-Sync 4) is installed on your desktop PC, file transfer can be done automatically each time the computers are connected in ActiveSync.

To transfer files “manually” proceed as follows. **NOTE THAT MICROSOFT ACTIVESYNC IS NEEDED TO CONNECT THE COMPUTERS even with an In-Situ synchronization utility installed (Win-Situ Sync or Pocket-Sync 4).**

*TIP:* Install Microsoft ActiveSync on the desktop PC before the first connection to the PDA. Be sure to exit your last Pocket-Situ session. The following procedure is performed from the desktop PC side.

1. With the computers connected and ActiveSync running, open up Explore on the ActiveSync tool bar.

2. Double-click on “My Pocket PC.”

3. Browse to the folder where Pocket-Situ Data is stored. On some PDAs this will be in a folder called “Storage Card” or “SD Card.” On the RuggedReader, look in Built-in Storage. Open the Pocket-Situ Data folder (double-click) to display a listing of your extracted data (.bin) files.

4. Right-click the file(s) you want to transfer and select Copy or Cut. Selecting Cut will remove the data file from the PDA.

5. Open My Computer or Windows Explorer for the desktop PC and navigate to the folder where you want to place the data file(s). When the cursor is in the desired folder, right-click, and select Paste.

We recommend placing the files in the following locations to ensure that they will be displayed properly in the Data Folder branch of the Win-Situ Navigation tree. The In-Situ synchronization utility, if used, will put the files in the proper locations automatically.

```
Test data files  Data folder in the folder where Win-Situ 4 is installed (normally, C:\Win-Situ\Data)
Low flow files  LowFlow subfolder of the Data folder
Calibration reports  Calibration Reports folder in the folder where Win-Situ 4 is installed. These will be accessible from Win-Situ’s Tools menu.
```
Logged data sets from the Multi-Parameter TROLL 9500 are called “tests.” Tests reside in the instrument until you extract them to the host computer. Any test that has been extracted can be viewed in text or graphical format. A test can be extracted and viewed at any time, even while it or another test is running.

The software provides two routes to view test data:

• If the test is in the instrument’s memory, extract it and select the View option at the end of the download. This launches the data viewer.

• If the test has been extracted to the host computer, it will appear in the Data Folder node in the Navigation tree. A connection to the instrument is not required in order to view extracted data.

SELECTING DATA IN THE DATA FOLDER

The Data Folder appears below the Home site in the Navigation tree. After data files have been extracted from the MP TROLL 9500 to the Data Folder, the node can be expanded to show device type, serial number, and extracted data files.

To view extracted test data:

1. Click the + sign beside the Data Folder to expand the listings.

2. The Data Folder has a node for data from each device type. TROLL 9500 data will be listed under the TROLL 9000 “family” device. Click the + beside the TROLL 9000 to expand the listing.

3. Device nodes are further subdivided by serial number. If tests from multiple TROLL 9500s have been extracted, the serial number of each will be displayed. Click the + beside the the serial number of the unit whose data you want to view.

4. The serial number node contains the extracted tests and logged Profiler data. Click to select the test you want to view.
The selected test is displayed in the Information pane—to the right of the screen in Win-Situ, or at the bottom in Pocket-Situ.

**Win-Situ Graphing Controls**

The Graph button displays data from the first four test channels in graph format. Time is shown on the X axis in the currently selected unit. The axes are auto-ranged. Click any graph to select it for formatting.

Each graph can display one, two, or all test channels. To change the appearance and content of any graph, display the Graph menu or right-click a graph. This provides access to the graph formatting options:

- change or add channels
- add data point indicators
- add grid lines to one or both axes
- add a logarithmic time scale
- change the range of the Y (data) axis
- change the parameter units
- add a time stamp to the X (time) axis
- zoom in on the graph

Many of these options can be set in the Graph settings window:

**TIP:** You can easily switch from Graph view to Report view and back using the Graph and Report buttons in the Information pane. However, the view chosen in this way is not “persistent” into your next session. To change the view so it always comes up as Graph or Report, specify the desired Data File View in Preferences (Options Menu) in Win-Situ or Setup in Pocket-Situ.

Win-Situ provides a range of options for viewing data in graph format. Here are some things you may wish to try:

- To enlarge the selected graph: Click Full Screen in the toolbar, then go to the Graph menu and select a different Number of Graphs
- To show two channels on the graph: select Graph Settings on the Graph menu, select a Primary channel and a Secondary Channel
- To show all channels on the graph: select Graph Settings on the Graph menu, choose All Channels Selected

**TIP:** The data file may appear in Report view or Graph view, depending on the preferences saved in your last Win-Situ or Pocket-Situ session.

5. To view the test data below the header in Report view:
   - Use the vertical and horizontal scrollbars to scroll through the data file.
   - Or click Full Screen in the shortcut bar to enlarge the text display, and then use the scrollbars.

**GRAPHING DATA**

6. Click or tap **Graph** to display the individual parameter data in graph format. See the sidebar on this page for Win-Situ graphing controls, and on the next page for Pocket-Situ graphing controls.

**TIP:** You can easily switch from Graph view to Report view and back using the **Graph** and **Report** buttons in the Information pane. However, the view chosen in this way is not “persistent” into your next session. To change the view so it always comes up as Graph or Report, specify the desired Data File View in Preferences (Options Menu) in Win-Situ or Setup in Pocket-Situ.
Pocket-Situ Graphing Controls

Pocket-Situ displays all the test channels in one graph. Time is shown on the X axis in the currently selected unit. The axes are auto-ranged.

The left Y axis represents the first channel in the test (usually temperature), or the first channel selected in the View... option in the command bar. The right Y axis represents the second channel.

Tap Graph or Text in the command bar to change the view.

The graph area may be resized as follows:

With the stylus (or other suitable pointer), press on the line that separates the Navigation tree from the Information pane and drag it up or down to resize the screen.

The View... option in the command bar launches wizards to help you change the way test data are displayed.

• Select Channels to View: Tap Next, then tap a check box to show or hide a channel. Tap Finish to return to the graph. The Y axes will reflect your selection.

• Change Units and other preferences.

• Change Channel Settings: Redefine the display options for a pressure channel. Refer to Section 7 for details on the settings.

CHANGING THE DATA DISPLAY

The View button launches wizards to let you change the way test data are displayed.

• Select Channels to View (Report view): Tap Next, then tap a check box to show or hide a channel. Finish returns to the Report view.

• Change Units and other preferences.

• Change Channel Settings: Redefine the display options for a pressure channel. Refer to Section 7 for details on the settings.

TRANSFERRING DATA TO EXCEL FROM WIN-SITU 4


2. On the File menu, select Export to Excel. If Microsoft® Excel® is properly installed on your PC, the data file will open in an Excel spreadsheet.

PRINTING DATA IN WIN-SITU 4

To print the graph or report as currently displayed:

1. Select Graph or Report view as desired.

2. On the File menu, select Print graph or Print report.

Additional information is available from your printer documentation.

SAVING TEXT FILES FROM WIN-SITU 4


3. In the window that opens, supply a filename in the usual way; the report will automatically be given a .TXT file type.

This graph was created with the following controls: Number of Graphs: One Graph, All channels selected, Zoomed in on 11-22 minutes, Vertical grid.
**WHY MONITOR PRESSURE?**

Water level or pressure head readings are taken for a number of reasons. In aquifer characterization, this type of data will help the user determine important hydrologic parameters of an aquifer, including hydraulic conductivity, transmissivity, permeability, storage coefficient, dispersivity, and porosity. Some or all of these parameters are important in the design of wells and well fields for various purposes.

In the design of a remediation system, the proper placement of wells for monitoring or extraction is of paramount importance if the system is to function effectively. Accurate data from slug and pump testing is one of the primary tools employed for aquifer characterization in the design process.

Step testing and constant-rate pump testing yield significant data in determining proper pumping rates. Improper rates can lead to aquifer depletion, salt water intrusion, and several other problems. Accurate data in designing the system and monitoring the system is essential for long-term success of the well and the proper maintenance of the aquifer.

The collection and use of water level measurements in the mining and coal bed methane industries are essential for the success of the mines and wells. Data derived from pilot testing of water wells constructed for coal bed methane extraction provide necessary information on bottom-hole pressure and appropriate pumping rates to accomplish the de-watering necessary to release the methane gas. Monitoring of water levels assures efficient production.

Water level measurements of surface water by pressure measurements yield significant data about the overall health of the hydrologic cycle. This information is also useful to government, agriculture, and industry as well as the scientific community when planning water use and when adjudicating water rights issues.

**THE PRESSURE SENSOR**

The optional pressure sensor of the Multi-Parameter TROLL 9500, if included, is permanently-installed and factory-calibrated. If your MP TROLL 9500 was ordered without a pressure or turbidity sensor, there will be a permanently installed plug in the pressure sensor slot. A pressure sensor can be added at the factory.

*Do not try to remove the pressure sensor or permanently installed plug.*

**FACTORY CALIBRATION**

The pressure sensor is calibrated across full pressure over a range of -5° to 50°C. The pressure reference is provided by a pressure control and measurement system (PCMS) that supplies a calibrated and certified pressure to the pressure sensor. The temperature reference is provided by a water bath with a thermal homogeneity of 0.0008 °C and measured with calibrated and certified digital thermometers. The calibration process results in a data set of raw analog to digital conversions (A/D readings) for both pressure and temperature from the device versus actual pressure and actual temperature from the calibrated references.

From a two-dimensional data set of temperature A/D versus reference temperature, and a three-dimensional data set of pressure A/D versus temperature A/D and reference pressure, numerical coefficients are generated to equations that map the MP TROLL performance across all temperatures and pressures.
**OPERATING PRINCIPLE**

A pressure transducer senses changes in pressure, measured in force per square unit of surface area, exerted by a column of water or other fluid above an internal media-isolated strain gauge. Common measurement units are pounds per square inch (psi) or newtons per square meter (Pascals).

**NON-VENTED (ABSOLUTE) VS. VENTED (GAUGED) SENSORS**

A non-vented or “absolute” pressure sensor measures all pressure forces detected by the strain gauge, including atmospheric pressure. Its units are psia (pounds per square inch “absolute”), measured with respect to zero pressure. Absolute pressure sensors are sometimes called “sealed gauge” sensors.

Absolute pressure measurements are useful during vacuum testing, in very deep aquifers where the effects of atmospheric pressure are negligible, and in unconfined aquifers that are open to the atmosphere.

With vented or “gauged” pressure sensors, a vent tube in the cable applies atmospheric pressure to the back of the strain gauge. The basic unit for vented measurements is psig (pounds per square inch “gauge”), measured with respect to atmospheric pressure. PSIG sensors thus exclude the atmospheric or barometric pressure component.

This difference between absolute and gauged measurements may be represented by a simple equation:

\[ P_{\text{gauge}} = P_{\text{absolute}} - P_{\text{atmosphere}} \]

If your MP TROLL 9500 includes a pressure sensor, it is either absolute or gauged. The pressure sensor type is not software-selectable.

However, psia measurements from absolute pressure sensors can be readily compensated for atmospheric pressure in the software due to the presence of the MP TROLL 9500’s onboard barometric pressure sensor, as long as the instrument is connected to vented cable. See “Correcting Absolute Pressure Readings for Barometric Pressure” later in this section.

**PRESSURE VS. DEPTH VS. LEVEL**

Display options for pressure measurements are completely software-selectable. Pressure sensor data may be displayed as raw pressure head, as depth, or as water level with a reference. When choosing depth or level, the software presents additional options for converting from pressure readings (in psi) to depth or level (in feet or meters), including a very accurate conversion that compensates pressure readings for fluid density, altitude, and latitude.

---

**ZEROING THE PRESSURE SENSOR**

The following procedure may be used, with caution, to “zero” the offset of a pressure sensor to correct for electronic drift. The drifted offset is visible when the sensor is in air and reading other than zero.

It is recommended you do not zero the offset if it is outside the specified accuracy of your pressure sensor, as shown in the table below. If the reading in air deviates from zero by more than the amounts shown, you may want to consider a factory recalibration. See the Appendix for additional information on electronic drift.

<table>
<thead>
<tr>
<th>Sensor range</th>
<th>Accuracy (%) full scale</th>
<th>Acceptable Offset from zero</th>
</tr>
</thead>
<tbody>
<tr>
<td>15 psig</td>
<td>± 0.05% FS</td>
<td>± 0.0075 psig</td>
</tr>
<tr>
<td>30 psig</td>
<td>± 0.05% FS</td>
<td>± 0.015 psig</td>
</tr>
<tr>
<td>100 psig</td>
<td>± 0.05% FS</td>
<td>± 0.05 psig</td>
</tr>
<tr>
<td>300 psig</td>
<td>± 0.05% FS</td>
<td>± 0.15 psig</td>
</tr>
</tbody>
</table>

1. With the MP TROLL connected to a host PC and selected in the Navigation tree, click or tap the Pressure parameter in the Parameters list.

2. Click or tap Calibrate. You will be prompted to ensure the device is in air.

3. When the device is in air, click Yes.

The current pressure reading will be set to zero. To check this, take a reading with the “Read” button.
SECTION 7: MONITORING PRESSURE (WATER LEVEL)

SETTING UP PRESSURE MEASUREMENTS: THE PARAMETER WIZARD

Win-Situ’s Parameter Wizard can help you configure a pressure channel to display measurements just as you want them. The specified settings are easily changed from one mode to another, and any choice can be redone or undone later, when viewing test data.

1. With the MP TROLL connected to a host PC and selected in the Navigation tree, select the pressure parameter.

2. Click or tap Edit.... The Parameter Wizard starts. The choices are discussed below.

3. When you finish the Wizard, Win-Situ sends the information to the device and updates the display.

PRESSURE SETUP CHOICES

Win-Situ prompts for the following when editing a pressure channel. You may not see all of these depending on your early choices.

1. NAME (OPTIONAL)

The pressure channel name is displayed in the Information pane and in test data files. A default name is proposed. If you choose, you may enter a custom name (up to 16 characters).

2. DISPLAY MODE

Select one of the following. Subsequent screens will be tailored to your choice.

- **Pressure Head** displays the raw pressure exerted by the column of water above the pressure sensor, in kiloPascals or pounds per square inch (psi). If you choose this mode, click Finish to end the Wizard. Then select units on Win-Situ’s Options menu.

- **Depth** converts the pressure of the water column to a depth reading, in meters, centimeters, feet, or inches. If you choose this mode, you will be asked to choose a method for converting pressure to depth. Then select units on Win-Situ’s Options menu.

- **Level - Surface**: Commonly used for surface water elevations. Data file readings are “positive up.” Increasing water levels will result in increasing readings. Decreasing water levels correspond to decreasing readings.

- **Level - Top Of Casing**: Commonly used for drawdown in groundwater wells. This mode is “positive down.” Decreasing water levels correspond to increasing readings, because the water level is getting further from the top of the well casing. Increasing water levels result in decreasing readings.

**Q:** What do I get with out-of-the-box pressure measurements (no setup)?

**A:**

The pressure channel defaults in Win-Situ and Pocket-Situ result in the following measurements, with no setup:

- Pressure head is measured
- Default units are psi
- No conversion from psi to depth/level

Remember that all these settings can be changed quite easily when you view the data after the test.
3. CONVERTING PRESSURE TO DEPTH OR LEVEL

The conversion from pressure in pounds per square inch (psi) to depth or level in meters or feet requires some knowledge of the properties of the fluid being monitored and optionally of the device’s location on the earth’s surface.

Conversion Using Specific Gravity

Pressure may be adequately converted to feet or meters of fluid using a dimensionless specific gravity value. A specific gravity of 1.0 (characteristic of pure water at 4°C) is adequate for many groundwater applications.

Conversion Using Density, Latitude, and Elevation

Pressure in the English unit psi is first converted to the SI unit Pascal (Pa). Conversion from Pascals to meters or feet requires two additional values:

- **Liquid Density.** Pure groundwater at 20°C has a density of 0.998 g/cm³. A table of density by temperature appears on this page. The Appendix includes a method to experimentally determine liquid density if the working fluid is not pure water.

- **Gravitational Acceleration.** The acceleration due to gravity that an object experiences is location-specific. Given your latitude and elevation, Win-Situ can calculate a value for gravitational acceleration for your location.

  **TIP:** Either specific gravity or density, latitude & elevation may be used to convert from pressure (psi) to depth/level (meters or feet). A gain in accuracy may be realized by selecting the density, latitude & elevation option.

4. LEVEL REFERENCE

If you selected a level display mode (Surface or Top of Casing), you will be prompted to enter a level reference. This is a user-specified starting point for level display modes, and can be any value you choose. Here are some examples:

- **Surface:** If you calculate the water level above mean sea level (MSL) and enter this as the Level Reference, then data will be displayed as elevations above MSL.

- **Top of Casing:** If you measure the distance to the water surface (DTW) from the top of the well casing and enter this number as the Level Reference, then data will be displayed as DTW values.

- A Level Reference of 0 is equivalent to “zeroing” the probe. Data will be displayed as changes, either positive or negative, from the starting water level.

Reference Time

The MP TROLL takes a “snapshot” of the sensor’s raw pressure reading, then your Level Reference is substituted. You can specify that the snapshot be taken now or when the test starts. The data file will show the Reference (raw pressure) Reading and when it was taken.

- **Now:** the pressure is measured when you finish setting up the pressure parameter, and the value is stored until the test starts. All measurements taken during the test will be relative to the water level at the time you finished the wizard.

- **Start of Test:** the Reference measurement will be taken at time t=0. All measurements taken during the test will be relative to the water level at the moment the test starts.

  **TIP:** After setting up the pressure channel with the desired display options, select the units you want to see. Use Win-Situ’s Options menu, or Pocket-Situ’s Setup button.

### Liquid Density

The conversion from PSI units to meters or feet of fluid requires several conversion factors. One of these is the density (ρ) of the aqueous solution being monitored. Pure groundwater at 20°C has a density of 0.998 grams per cubic centimeter (g/cm³).

Use the values in the table for density if you do not otherwise know it. However, since these data assume pure water, there is no accommodation for other variables (such as salinity) that can affect your actual water conditions. Alternatively, you could compute the fluid density using the procedure given in the Appendix.

**Fluid density by temperature**

<table>
<thead>
<tr>
<th>Temp. (°C)</th>
<th>Density (g/cm³)</th>
<th>Temp. (°C)</th>
<th>Density (g/cm³)</th>
<th>Temp. (°C)</th>
<th>Density (g/cm³)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.999900</td>
<td>11</td>
<td>0.999605</td>
<td>21</td>
<td>0.997992</td>
</tr>
<tr>
<td>2</td>
<td>0.999941</td>
<td>12</td>
<td>0.999498</td>
<td>22</td>
<td>0.997770</td>
</tr>
<tr>
<td>3</td>
<td>0.999965</td>
<td>13</td>
<td>0.999377</td>
<td>23</td>
<td>0.997538</td>
</tr>
<tr>
<td>4</td>
<td>0.999973</td>
<td>14</td>
<td>0.999244</td>
<td>24</td>
<td>0.997296</td>
</tr>
<tr>
<td>5</td>
<td>0.999965</td>
<td>15</td>
<td>0.999099</td>
<td>25</td>
<td>0.997044</td>
</tr>
<tr>
<td>6</td>
<td>0.999941</td>
<td>16</td>
<td>0.998943</td>
<td>26</td>
<td>0.996783</td>
</tr>
<tr>
<td>7</td>
<td>0.999902</td>
<td>17</td>
<td>0.998774</td>
<td>27</td>
<td>0.996512</td>
</tr>
<tr>
<td>8</td>
<td>0.999849</td>
<td>18</td>
<td>0.998595</td>
<td>28</td>
<td>0.996232</td>
</tr>
<tr>
<td>9</td>
<td>0.999781</td>
<td>19</td>
<td>0.998405</td>
<td>29</td>
<td>0.995944</td>
</tr>
<tr>
<td>10</td>
<td>0.999700</td>
<td>20</td>
<td>0.998203</td>
<td>30</td>
<td>0.995646</td>
</tr>
</tbody>
</table>
QUICK SUMMARY OF PRESSURE SETUP

For DEPTH:

1 Select Pressure in Parameters list, select Edit

2 Display Mode: Select Depth, click Next.

3 Select Depth/Level Conversion: Specific Gravity 1.0. Click Finish.

For AQUIFER TESTING:

1 Select Pressure in Parameters list, select Edit

   Display Mode
   Depth/Level Conversion Specific Gravity 1.0

2 Display Mode: Select Level - Top of Casing, click Next.

3 Select Depth/Level Conversion: Specific Gravity 1.0. Click Next.

4 Select Level Reference 0 & Reference Reading Start of Test. Click Finish.

For SURFACE WATER MONITORING (install probe before setting these):

Display Mode       Level - Surface
Depth/Level Conversion Specific Gravity 1.0
Level Reference     Current elevation or gauge height of water surface
Reference Reading   Now (with probe installed)

After setting the pressure mode, select the UNITS you want:

Win-Situ: Options menu > Preferences, scroll down to LEVEL/DEPTH, select unit, click OK

Pocket-Situ: Tap Home at top of screen, tap Setup at bottom of screen, scroll down to LEVEL/DEPTH, select unit, click OK
INSTALLATION FOR PRESSURE/LEVEL MEASUREMENT

Position the instrument below the lowest anticipated water level, but not so low that its range might be exceeded at the highest anticipated level. Lower the MP TROLL 9500 gently to the desired depth.

If real-time readings are not required, remove the TROLL Com from the cable after programming. Protect the “uphole” end of the cable with the dust cap or optional desiccant. If cable will not be used, attach a Twist-Lock Hanger to the TROLL 9500.

SECURING THE CABLE

The RuggedCable has a handy device called a Kellems® grip near the surface end. You can slide it along the cable to the desired position by compressing it. When you pull on it, it tightens and stops sliding. You may need to pull on both ends of the Kellems grip to properly tighten it and keep it from slipping.

Use the loop of the Kellems grip to anchor the cable to a convenient stationary object. It works well with In-Situ’s “well dock” installation ring. Simply insert the loop into the locking clip on the well dock, and position the assembly on the top of a well. Insure the cable is secured to prevent the instrument moving while data is being logged.

STABILIZATION TIME

Allow the instrument to stabilize to the water conditions for about an hour before starting a test. A generous stabilization time is always desirable. Even though the cable is shielded, temperature stabilization, stretching, and unkinking can cause apparent changes in the probe reading. If you expect to monitor water levels to the accuracy of the probe, it’s worth allowing the extra time for the probe to stabilize to the test environment.

<table>
<thead>
<tr>
<th>Range</th>
<th>Usable Depth</th>
</tr>
</thead>
<tbody>
<tr>
<td>kPa</td>
<td>PSI</td>
</tr>
<tr>
<td>103.4</td>
<td>15</td>
</tr>
<tr>
<td>206.8</td>
<td>30</td>
</tr>
<tr>
<td>689.5</td>
<td>100</td>
</tr>
<tr>
<td>2068</td>
<td>300</td>
</tr>
</tbody>
</table>

TWIST-LOCK HANGER INSTALLATIONS

The non-vented Twist-Lock hanger accessory can be used to suspend a pre-programmed MP TROLL 9500 in a well or other site while taking data where barometric pressure changes are not crucial.

The Twist-Lock hanger allows use of inexpensive hanging cable, and requires no direct communication and no cable venting. This setup is ideal for use where barometric compensation of pressure measurements is not required—in vacuum testing, unconfined aquifers, or very deep applications where barometric pressure effects are minimal.

- Because the Twist-Lock Hanger has no communication capabilities, you must program the MP TROLL 9500 before installation.
- Logged pressure data will show the effects of changes in barometric pressure. However, post-processing tools may be used to eliminate the effects of barometric pressure changes from the data, if required. See "Correcting Absolute Pressure Readings for Barometric Pressure" below for more information.

Installation Tips for Level/Depth/Pressure Monitoring

- Never let a probe “free fall” down a well. The resulting shock wave when it hits the water surface can damage the pressure sensor strain gauge (the “waterhammer” effect), as well as other sensors.
- It is always wise to check the level of water above the probe, then move it and read again to be sure that the probe is giving a reasonable reading and showing change. It might not be located where you think it is — for example, it could be wedged against the casing with a loop of cable hanging below it. A probe in such a position might become dislodged and move during the test, giving a false change in level. A secure placement is critical to accurate level measurements.
- When monitoring pressure with vented cable, do not allow the cable (and its internal vent tube) to kink or bend. If the vent tube is obstructed, water level measurements can be adversely affected. The vent tube can become kinked and damage the internal components without any visible harm to the outside of the cable. The recommended minimum bend radius is 63.4 mm (about 2½ in) or more.
- Do not position the instrument below the level of the pump in a pumping well. The pressure transients generated by the pump will cause false level readings. Large pumps can swallow the probe and cause permanent damage to both the MP TROLL 9500 and the pump.
**CORRECTING ABSOLUTE PRESSURE READINGS FOR BAROMETRIC PRESSURE**

Pressure measured with an “absolute” or “non-vented” (psia) pressure sensor includes all pressure forces detected by the strain gauge—atmospheric pressure as well as the pressure due to the water column. “Gauged” or “vented” (psig) sensors remove the atmospheric pressure component (pressure due to the atmosphere), so that atmospheric pressure on a water surface is not doubled in the measurements from a sensor under the water surface.

Barometric compensation of absolute pressure measurements, if required, may be accomplished in a couple of ways.

**MANUAL BAROMETRIC PRESSURE COMPENSATION**

**Using the MP TROLL’s Barometric Pressure Sensor**

If your model of MP TROLL 9500 includes an onboard barometric pressure sensor, and the instrument is attached to vented cable, the barometric pressure sensor output can be used to correct measurements from a submerged absolute (psia) pressure sensor. In this case, set up the pressure channel in “pressure head” mode, and select the same units for both the water pressure channel and the barometric pressure channel. Set up and run a test that includes both pressure channels. Extract (download) the data, and subtract the barometric pressure measurements from the water pressure measurements.

**Using a Second Unit and a Spreadsheet**

If a barometric pressure sensor is not present, and/or the cable is not vented, barometric compensation of absolute pressure measurements may be accomplished with the use of a separate device—a Baro TROLL or Absolute miniTROLL—installed above the water surface, taking barometric pressure measurements. Set up both devices to use the same units and display mode—the only difference will be if a level reference is used; in that case the barometric reference should be zero, the downhole reference will be the water level. Schedule tests to start at the same time in each unit. Extract (download) the data from both tests, and subtract the barometric pressure measurements from the water pressure measurements (by hand or in a spreadsheet).

**TIP:** For additional information on correcting absolute pressure measurements, see the Technical Note “Manual Barometric Corrections” in the Downloads section of our website at www.in-situ.com.

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**AUTOMATIC BAROMETRIC PRESSURE COMPENSATION**

A Baro Wizard in Win-Situ and Pocket-Situ allows for automatic barometric pressure compensation of absolute pressure data, either using a fixed user-specified value or by reference to a file of barometric pressure data collected at the same time by a BaroTROLL or other absolute (psia) instrument installed at the surface. The barometrically corrected absolute pressure data may be saved in a new file, if desired.

**Collecting Barometric Pressure Data for Reference**

Be sure to set the clock in the BaroTROLL (or other absolute instrument). Add a test. Ideally, this test should start before the test in the Absolute MP TROLL 9500 starts and log data on approximately the same schedule. Run the test. Extract the test (it is not necessary to stop it.)

**Collecting PSIA Data**

Set the clock in the Absolute MP TROLL 9500. Add a test and run it as usual. Extract this test also.

**Using the Baro Wizard**

The difference in pressure between a BaroTROLL at the surface and a submerged MP TROLL 9500 with an absolute (psia) pressure sensor can be calculated from extracted test data files. This task is simplified if the BaroTROLL test covers the entire duration of the Absolute instrument’s test.

1. Launch Win-Situ 4 or Pocket-Situ 4. Connection to an instrument is not necessary after the tests have been extracted.
2. In the data folder, select a test extracted from an MP TROLL 9500 with an Absolute pressure sensor (PSIA data).
3. On the Tools menu, select Baro Wiz.
4. In the Barometric Correction window, select the type of correction to be performed:
   - None
   - Value: Enter a barometric pressure value to be subtracted from all data points in the test file. Units are selectable, and your entry is checked for validity. For example, in inches of mercury the valid range is 14.3 to 33.5 (covering altitudes from the Alps to the Dead Sea). You may not leave this entry blank, because a fixed value will be needed even with a reference file (next option, see below) in case the time stamps in the two files do not completely overlap.
   - File: Select a test of barometric pressure data collected by a BaroTROLL or other PSIA instrument. This becomes the Reference file from which barometric pressure corrections will be derived. If the time stamps in the files do not overlap completely, the fixed value will be applied.

5. To save the corrected data in a new file that will be automatically named, check the create new file checkbox.

6. Click OK. The correction is performed immediately and the corrected file is displayed.

**RECALIBRATION RECOMMENDATIONS**

Pressure sensor accuracy can be adversely affected by improper care and handling, lightning strikes and similar surges, exceeding operating temperature and pressure limits, physical damage or abuse, as well as normal drift in the device's electronic components. Aside from damage to the sensor, the need for factory recalibration is dependent upon the amount of drift a customer is willing to tolerate. Factory calibration every 12-18 months is recommended. Contact In-Situ Customer Service for information on the factory maintenance and calibration plan. Calculations of the accuracy drift of the MP TROLL 9500 over time are contained in the Appendix of this manual. Contact In-Situ Customer Service for information on periodic check-ups and recalibration.
8 MONITORING TEMPERATURE

WHY MONITOR TEMPERATURE?

Water temperature plays an important role in water chemistry, which in turn influences the biological activity and growth of aquatic organisms. In general, the higher the water temperature, the greater the biological activity and the rate of chemical reactions. An important example of the effects of temperature on water chemistry is its impact on oxygen. Warm water holds less oxygen than cool water; the maximum amount of oxygen that can be dissolved in the water decreases as water temperature increases.

Artificially high temperatures are often referred to as “thermal pollution,” which may result from discharge of municipal or industrial effluents. Thermal pollution can have a significant ecological impact. In running waters, particularly small urban streams, elevated temperatures from road and parking lot runoff can be a serious problem for populations of cool or cold-water fish.

Changes in the growth rates of cold-blooded aquatic organisms and many biochemical reaction rates can often be approximated by the “Q₁₀ rule,” which predicts that growth rate will double if temperature increases by 10°C (18°F) within their “preferred” range.

Knowledge of water temperature is essential to the measurement of dissolved oxygen, conductivity (salinity), pH, and many other water-quality parameters. In limnological studies, water temperature as a function of depth can be an important indicator. Industrial plants often require data on water temperature for process use or heat-transmission calculations.

These are only a few of many reasons for measuring and recording water temperature over the short or long term.

THE TEMPERATURE SENSOR

All models of the Multi-Parameter TROLL 9500 include a permanently installed, factory-calibrated sensor for measuring solution temperature.

**TIP:** After a drop or shock, if the end of the temperature sensor is slightly bent, it may be carefully bent by hand (no tools, please!) back to its original alignment. Note that a severe shock may affect the accuracy of the temperature sensor.

The MP TROLL 9500 temperature sensor is the Standard Platinum Resistance Thermometer specified by the ITS-90 (International Temperature Scale of 1990). A platinum resistance thermometer (PRT) is a type of resistance temperature detector (RTD).

End view of sensor block

Platinum Resistance Thermometer

Knowledge of water temperature is essential to the measurement of dissolved oxygen, conductivity (salinity), pH, and many other water-quality parameters. In limnological studies, water temperature as a function of depth can be an important indicator. Industrial plants often require data on water temperature for process use or heat-transmission calculations.

These are only a few of many reasons for measuring and recording water temperature over the short or long term.
ROLE OF THE SENSOR IN CALIBRATION

The temperature sensor has a primary function during calibration of temperature-dependent water-quality parameters such as conductivity. When the calibration temperature is known, temperature compensation can be provided during measurement of water-quality parameters. The temperature sensor also provides continuously updated real-time measurements of solution temperature when profiling.

Ensure that the temperature sensor is immersed in at least one-half inch of solution during calibration of all parameters. The fill line on the Cal Cup serves as a guide to the recommended quantity of calibration solution.

For best results, the water-quality sensors should be calibrated at the same temperature that will be encountered in the field.

SOFTWARE FUNCTIONS

Conversion of resistance (measured by the PRT) to temperature is automatic in the software. No user calibration is required.

The temperature channel is automatically included in tests so that solution temperature is available to compensate water-quality data from other channels.

Units

Temperature may be displayed in degrees Celsius (°C) or degrees Fahrenheit (°F).

TIP: To change unit preferences: In Win-Situ, select Preferences on the Options menu. In Pocket-Situ, select the Home site, then tap Setup in the command bar.

REFERENCES


Water on the Web (WOW). University of Minnesota project initially funded by the National Science Foundation. On the web at wow.nrrri.umn.edu.
9  MONITORING BAROMETRIC PRESSURE

WHAT IS BAROMETRIC PRESSURE?
Barometric, or atmospheric, pressure is the force exerted by the gases in the atmosphere everywhere on the surface of the earth. Barometric pressure is greatest at or below sea level, and decreases as altitude above sea level increases.

WHY MONITOR BAROMETRIC PRESSURE?
Barometric pressure influences the measurement of water levels in water bodies that are open to the atmosphere. It also determines the amount of atmospheric gases that can be dissolved in water; more oxygen, for example, can be dissolved in water at higher barometric pressure (lower altitude). Barometric pressure additionally influences other water-quality parameters such as pH.

A logged record of barometric pressure data during a test can be used to correct water level measurements made with an absolute pressure sensor to factor out the effects of barometric pressure fluctuations.

To measure barometric pressure, the probe must be vented to the atmosphere. A submerged Multi-Parameter TROLL on non-vented cable cannot accurately report barometric pressure.

If your MP TROLL 9500 is used with a non-vented backshell and/or non-vented cable, a software correction can substitute for barometric pressure venting. See the procedure in this section.

THE BAROMETRIC PRESSURE SENSOR
Most models of the Multi-Parameter TROLL 9500 include a permanent, factory-calibrated internal barometric pressure sensor. Its primary function is in calibration and measurement of water-quality parameters, such as dissolved oxygen, that are dependent upon barometric pressure. It may also be used to compensate fluid pressure measurements made with an absolute pressure sensor.

The sensor is automatically included in tests so that its value is available to compensate water-quality data from other channels.

Units
The following units are available for barometric pressure: Bars, milliBars, inches of mercury (in. Hg or “ Hg), millimeters of mercury (mm Hg), cm, pounds per square inch (psi)

TIP: To change unit preferences: In Win-Situ, select Preferences on the Options menu. In Pocket-Situ, select the Home site, then tap Setup in the command bar.

COLLECTING ACCURATE MEASUREMENTS WITHOUT VENTED CABLE
Win-Situ and Pocket-Situ cannot determine whether the MP TROLL 9500 is on vented or non-vented cable. However, you can supply the software with this information, and also enter a fixed barometric pressure value to be used in the absence of cable venting.

Procedure:
1. Select the barometric channel in the Navigation tree.
2. Click or tap Edit.... The Edit Barometric Channel screen appears. Several options are presented:

TIP: This screen can also be accessed during test setup and dissolved oxygen calibration.
3. Do one of the following:

- If this MP TROLL 9500 has a barometric pressure sensor and **vented** cable, select “Use vented cable.” Go to step 5.
- If the device is on **non-vented** cable now and will take measurements using **non-vented** cable, select “Use non-vented cable or backshell.” Continue to step 4.
- If the TROLL is on **vented** cable now but will take measurements on **non-vented** cable or backshell” and also check the last box so a reading can be taken from the barometric pressure sensor while it is available.

4. Supply a barometric pressure value for the software to use in the absence of vented cable. Choose one of these options:

- Supply a fixed pressure value in your choice of units (select units by clicking the arrow on the list box). This value will be used each time barometric pressure is required elsewhere in the software—for example, when calibrating dissolved oxygen.
- Enter your elevation and let the software calculate barometric pressure based on the values in the table on this page. Please note that *this fixed value will not be recalculated if the elevation changes.*

5. Click **OK** to store this information.

### PRESSURE VS. ELEVATION
(based on U.S. Standard Atmosphere)

<table>
<thead>
<tr>
<th>Elevation</th>
<th>Pressure</th>
</tr>
</thead>
<tbody>
<tr>
<td>ft</td>
<td>m</td>
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<tr>
<td>16,500</td>
<td>5029.2</td>
</tr>
</tbody>
</table>

### Conversions
- bars * 29.530 = inches of mercury
- psi * 2.036 = inches of mercury
- atmosphere * 101325 = Pascals
- psi * 6.894757 = 10^3 = Pascals
- bars * 14.50337 = psi
10 MONITORING WATER QUALITY: OVERVIEW

WHY MONITOR WATER QUALITY?

At a time of increasing demands on the finite natural resources of our planet, public organizations and private individuals alike have become acutely aware of the responsibility to maintain the quality of the earth's air and water supplies.

Recent rapid advances in knowledge and technology have made it possible to deliver accurate, timely, and reliable data on processes we cannot necessarily examine visually. New-generation sensors for in-situ measurement of surface waters and groundwater can be an efficient alternative to time- and labor-intensive programs of field sampling and transportation to a laboratory for analysis, or can supplement such programs. If it is possible to collect, interpret, and respond in a timely fashion to accurate information about water supplies and water quality, we can design better systems for protection of those supplies.

Monitoring water-quality parameters can reveal much about the presence and movement of natural and unnatural components of water—the presence of harmful bacteria, potential pollution sources, depletion of nutrient requirements for aquatic life, salt-water intrusion into fresh water bodies, changes in water level or temperature that can alert observers to the onset of an “event” that can adversely affect the quality of the resource.

Monitoring water quality in surface and groundwater resources may be required by Federal, state, or local regulations. Digital records of monitoring can document compliance with guidelines and standards mandated by regulatory bodies.

Profiling and logging water-quality data can provide timely information on continually changing conditions—profiling to provide instantaneous real-time feedback, logging to track trends and demonstrate compliance.

THE SENSORS

The Multi-Parameter TROLL 9500 takes advantage of new technologies to monitor water-quality parameters in-situ with high accuracy. Each sensor has been manufactured to our rigid specifications and is designed to operate with the entire suite of sensors and with the MP TROLL 9500 electronics. These “smart” sensors retain serial number identification and calibration information, and are detected and identified by the instrument. A sensor may be calibrated in any MP TROLL 9500 and moved to another port that accepts the sensor type, or used in another MP TROLL 9500, without recalibration. The most accurate results will be obtained when a sensor is calibrated and operated in the same MP TROLL 9500.

The water quality sensors available for the Multi-Parameter TROLL 9500 may be classed in two general types:

BASIC SENSOR SET

- pH
- Combination pH/ORP (Oxidation-Reduction Potential)
- Dissolved Oxygen, polarographic (DO)
- Conductivity (and Specific Conductance, Salinity, Total Dissolved Solids, Resistivity)

The pH sensor is a Single ISEs (ion-selective electrode). The Combination pH/ORP sensor is a Multiple ISE.

The Basic sensors can be factory-calibrated and pre-installed in the MP TROLL 9500. They are ready for use right out of the box with a brief Quick Cal. However, for best results, if your software supports it, we recommend that you perform a traditional two-point calibration for pH and DO, and a specific range calibration for conductivity as described in sections 11-13 below. The accuracy that can be achieved from the instrument is proportional to the time and care you put into calibration.
**EXTENDED SENSOR SET**

The Extended Sensor set includes:
- Ammonium
- Chloride
- Nitrate
- Turbidity (factory-installed)
- RDO<sup>®</sup> Optical Dissolved Oxygen

**INSTALLING SENSORS**

The diagram below represents a head-on view of the “sensor block” in the front end of the MP TROLL 9500. There are four sensor ports, plus permanently installed pressure and/or turbidity and temperature sensors. Pressure and turbidity sensors are optional—if your instrument does not include one or both of these sensors, there will be a permanently installed plug in the port.

**Q:** Will a sensor work if installed in the wrong port?

**A:** Physically, a sensor may be plugged into any port. However, a sensor that is detected in the wrong port for its type will generate an error message in the software. The message will let you know which port or ports the incorrectly installed sensor should be moved to.

In this case, remove the offending sensor and install it in the correct port. Then “refresh” the device view in the software to update the display.

2. Remove the cap or storage bottle from the sensor. Retain the cap or bottle for future storage and protection of the sensor. If the connector end is covered with a cap, remove it also.

**TIP:** To ensure optimum membrane response for new ion-selective electrodes (pH, ORP, nitrate, ammonium, chloride), soak the sensor in calibration solution for at least 15 minutes and up to several days before calibration.

3. Remove any moisture or dirt from the area around the port where you will install the sensor, then use the sensor removal tool to remove the plug from the port. Retain the plug for use with fewer than 4 removable sensors installed.

**TIP:** If you are installing a sensor in port 3 (the central port), install it first. This will make it easier to install sensors in other ports.

4. Remove any moisture or debris from the connector in the bottom of the port with a clean swab or tissue.

5. Check lubrication of the sensor o-rings.

**TIP:** The sensor o-rings require generous lubrication before installation. New sensors will be lubricated at the factory. If the o-rings appear dry, apply a silicone lubricant before installation.

6. Align the mark on the sensor with the alignment mark on the correct port (see diagram), or visually align the sensor connector pins with the port connector pins.

7. Press the sensor firmly into the port until it is securely seated. When properly inserted a small gap (width of the sensor removal tool) remains between the instrument body and the widest part of the sensor, for ease of removal.
REMOVING SENSORS

Sensors may be removed for inspection, cleaning, routine maintenance, and storage. Because the smart sensors retain calibration information, they may be removed and re-installed—even in another MP TROLL 9500—as often as necessary.

Remove a sensor by positioning the yoke of the sensor removal tool at the point where the sensor enters the sensor block. Firmly pry the sensor upward until it pops out.

Sensor O-Rings

Two Viton® o-rings on each sensor provide a watertight seal against water leakage into the instrument body. We recommend that you inspect these o-rings each time you remove or install a sensor. Check carefully for cracks, tears, splitting, shredding, and other damage. If the o-rings are in good condition, apply silicone lubricant before installing the sensor again. Remove excess lubricant with a tissue, and take care to keep grease away from the area around the connector at the bottom of the sensor. Should lubricant get into this area, it can be removed with a clean cotton swab.

If the o-rings become damaged to the extent that no longer provide an effective seal, they should be replaced. Sensor o-rings and lubricant are available from In-Situ Inc. or your distributor.

CALIBRATION OVERVIEW

The MP TROLL 9500 and its control software provide several options for calibration of the water-quality sensors. Select the method that suits the time you have at your disposal and the degree of accuracy you want to achieve when measuring water-quality parameters.

Satisfactory results may be achieved using the Quick Cal procedure. Some sensors can even return nominal results straight out of the box using the factory-supplied default calibration coefficients. However, for best results we recommend a full traditional calibration procedure before the first field use, and periodic checks and recalibrations as necessary thereafter.

The following available options are briefly described in the next two pages:

• Traditional Calibration
• Quick Calibration
• Out-of-Box
• Factory Defaults

TRADITIONAL CALIBRATION

A full traditional calibration, guided by software wizards, can achieve the highest level of accuracy. Some sensors require a single-point calibration, others present a choice of single- or multi-point, requiring more than one calibration standard. A single-point calibration gives good results in the range of values represented by the selected calibration solution. When a wide range of values are expected, a multi-point calibration is recommended.

With the sensor installed in the MP TROLL 9000 and immersed in calibration solution, the sensor is powered at regular intervals and its response is monitored. The difference (deviation) between the minimum and maximum response over a predetermined time period is tracked by the software. When the peaks of the response fall within predetermined limits for the time period, the sensor response is considered sufficiently stable to provide a valid calibration point. The length of time and allowable deviation are specific to each sensor type, and furthermore are specific to the determination of nominal stability or complete stability. The time period of interest is shorter for nominal stability than for complete stability, allowing for a shortening of the calibration soak time while still returning a valid calibration point.

• Available for: All water-quality parameters.
• Requirements: MP TROLL 9500, sensors (installed), Cal Cup, and one or more calibration solutions for each parameter to be calibrated. Suitable calibration solutions are supplied in In-Situ’s individual calibration kits.
• Where to find the method in this manual: Sections 11-19.

Q: What is the difference between NOMINAL and STABLE?

A: To meet the criteria for a valid calibration point, the change (deviation) in sensor response is monitored over time. The software is looking for the calibration solution temperature and the sensor readings to settle over a specific time period. The criteria for STABLE are designed to meet the published specifications. The NOMINAL criteria are designed to shorten the calibration time when an approximate calibration is acceptable. When the deviation falls within the limits of the “loosened” specifications, NOMINAL is displayed in the Status area, and the Accept button becomes available to store the current calibration point.

Accepting a NOMINAL value may save considerable time. In some cases, especially if the sensors have been soaking in the solution for several minutes prior to calibration, the accuracy achieved by accepting a nominal value may be very similar to that obtained by waiting for complete stability.
QUICK CALIBRATION

A “Quick Cal” calibrates the Basic sensors simultaneously to achieve adequate performance with minimal labor using a single “universal” calibration solution.

- Available for: pH, ORP, polarographic Dissolved Oxygen, and Conductivity.
- Requirements: MP TROLL 9500, sensors (installed), Cal Cup, and Quick Cal solution.
- Where to find the method in this manual: Section 3, Getting Started.

OUT OF THE BOX

Some sensors may be installed and used right out of the box using factory-supplied default calibration coefficients.

- Available for: pH, ORP, and Conductivity.
- Requirements: MP TROLL 9500, sensors (installed).
- Where to find the method in this manual: No method required, plug-and-play.

DEFAULT COEFFICIENTS

This option resets the sensor’s factory defaults and is best when the sensor is new.

- Available for: pH, ORP, and Turbidity.
- Where to find the method in this manual: See pH calibration in Section 11, ORP calibration in Section 14, Turbidity calibration in Section 18.

The cell constant for a conductivity sensor may be entered “by hand,” without performing a complete calibration, if desired. See the procedure in Section 12 below.

TIP: When using Pocket-Situ to perform calibrations, do not let the PDA time out during the procedure. To locate this setting in most PDAs, display the Start menu, select Settings, System tab, Power.

PREPARING TO CALIBRATE

CALIBRATION KITS

Kits of calibration solutions for various parameters and ranges are available from In-Situ Inc. Our calibration solutions are certified to N.I.S.T. standards, packaged in quarts, each providing sufficient fluid for at least 6 calibrations. Kits include deionized water if that substance is recommended for rinsing a particular sensor during calibration.

The Quick Cal kit provides a convenient “universal” calibration solution, designed to calibrate multiple parameters simultaneously.

THE CALIBRATION CUP

The clear acrylic Cal Cup shipped with your MP TROLL 9500 is used to hold solution during sensor calibration. When fitted with a small moist sponge, it also provides a convenient way to protect and hydrate the sensors of the MP TROLL 9500 between uses.

The base of the Cal Cup is removable so that the stirrer may be attached for calibrations where continuous agitation of the solution is recommended. A small hole in the threads of the base near the o-ring permits venting during 100% dissolved oxygen calibration with the Cal Cup and probe inverted.

The Cal Cup’s fill lines indicate the recommended amount of solution for most calibrations, and ensures the temperature sensor is immersed.

- With a full complement of sensors installed, use the lower line as a guide.
- With only 1 or 2 removable sensors installed, fill to the upper line.

The temperature sensor should always be immersed in at least one-half inch of fluid.

When attaching the Cal Cup to the front end of the MP TROLL 9500, align carefully and thread the Cal Cup onto the body until seated against the o-ring, then back off slightly to avoid overtightening.

When attaching the Cal Cup to the instrument body, be careful not to overtighten.
EFFECT OF TEMPERATURE ON CALIBRATION
The most successful calibrations reproduce field conditions as nearly as possible, especially temperature. It is best to calibrate at the expected field temperature.

RINSING
As a general guideline, we recommend you rinse the Cal Cup, the front end of the MP TROLL 9500, and the installed sensors prior to beginning calibration. This will remove trace contaminants or solutions used in previous calibrations, and prepare the instrument for a clean calibration.

A good way to do this is to fill the Cal Cup with water, attach to the instrument, and shake vigorously. This may need to be done a couple of times.

Rinse first in tap water, followed by a rinse with distilled or deionized water.

Shake or wipe with a clean lint-free tissue to dry. It is not necessary to dry thoroughly.

Some calibration procedures also recommend a rinse in the selected calibration solution. In this case, drying is not necessary.

STIRRING
When to Stir?
The stirrer accessory should be used during a calibration procedure if it will also be used during field use—for example, if the instrument will be in stagnant or very slowly moving water. The more closely calibration conditions reflect field conditions, the more successful the calibration. This is especially important when calibrating the ISE sensors (ammonium, chloride, and nitrate).

ISE sensors in close proximity to each other can sometimes create interferences. Constant stirring can enhance the performance of the ISE sensors.

Attaching the Stirrer for Calibration
To use the battery-powered stirrer for calibration, attach it to the MP TROLL 9500 and Cal Cup as follows. See illustration C on the following page.

1. Remove the restrictor (nose cone attached) from the MP TROLL 9500 and set it aside.
2. Remove the black end cap from the Cal Cup.
3. Screw the top of the stirrer (propeller end) to the bottom of the Cal Cup (the end from which you just removed the end cap).
4. Fill Cal Cup to fill line with solution.
5. Attach Cal Cup/stirrer assembly to front end of MP TROLL 9500.

Starting the Stirrer
The stirrer is powered by two alkaline D-cells (installed). To start the motor, tighten the end cap.

The magnetic stir bar in the propeller compartment will start to spin. The stir bar is protected by a guard plate that may be removed for cleaning if necessary.

TIP: Should the stir bar not start spinning, try giving it a gentle nudge by sliding a narrow tool such as a screwdriver or key between the protective bars of the guard plate.

To turn the stirrer off, back off the end cap until the stir bar stops spinning.

CALIBRATION PROCEDURES
Refer to the following sections for specific calibration procedures and guidelines:
- Quick Cal Section 3
- pH Section 11
- Conductivity Section 12
- Dissolved Oxygen (polarographic) Section 13, first half
- Dissolved Oxygen (optical) Section 13, second half
- ORP Section 14
- Ammonium Section 15
- Chloride Section 16
- Nitrate Section 17
- Turbidity Section 18

AFTER CALIBRATION
When you finish calibrating with any method, the following happens:
- The newly calculated calibration coefficients are written to the “smart sensor” memory.
- You have the option of viewing the calibration report. The report may be viewed immediately after calibration, or at any time. See Calibration History, below.
- The sensors are ready to take measurements.
It is best to calibrate just before field use. However, should you need to store calibrated sensors, there are a couple of options:

- If the instrument will be used in a day or so, leave the sensors installed. Remove the Cal Cup and rinse it and the sensors. Moisten a sponge and place it in the bottom, or add a little water (deionized, distilled, or tap) to the Cal Cup—just enough to create a moist environment. Return the probe to the Cal Cup for transport to the field site.

  **TIP:** Deionized water is preferred over tap water, but it is not essential; especially if the local tap water is of good quality.

- For longer storage, remove the sensors from the MP TROLL 9500. Store the conductivity sensor dry. Store the DO, pH, and pH/ORP sensors in their storage bottles (located in the sensor kits): DO in clean water, pH and pH/ORP in the solution they were shipped in, or with a moist sponge in the sensor storage bottle to avoid depleting the reference solution.

**CALIBRATION HISTORY**

Each time a sensor is calibrated, the information is written to the sensor, where it is stored until the next calibration. Details on the most recent calibration are displayed by the software when a parameter is selected in the Navigation tree.

The software also creates a calibration report in html format each time a sensor is calibrated. A separate report is created for every calibration of every parameter—even for a calibration that was cancelled. You have the option to view the report immediately after calibration. Reports are stored for later retrieval and reference in a folder named “Calibration Reports” in the folder where Win-Situ 4 or Pocket-Situ 4 is installed. Reports include a detailed record of date and time, parameter, calibration type, number of calibration points, stimulus and response, and calculated coefficients. An index in html format is also created and updated each time a calibration is performed.
The calibration reports are accessible from the Tools Menu and the Show Calibration Report button on the toolbar. They may also be accessed like other files through Windows Explorer (desktop PC) or File Explorer (PDA); they are not displayed in the Data Folder. They may be viewed or printed to provide a complete calibration history.

**TIP:** Here’s how to find the calibration report indexes:

- Desktop or laptop PC—Calibration Reports subfolder in the folder where Win-Situ 4 is installed
- PDA—Calibration Reports subfolder in the folder where Pocket-Situ 4 is installed

**Example of a calibration report index**

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<tr>
<th>Folder</th>
<th>Click a folder to see its listings</th>
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<tbody>
<tr>
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</tr>
<tr>
<td>SN45025</td>
<td></td>
</tr>
</tbody>
</table>

**SN32072**

<table>
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<tr>
<td>SN32072 2006-04-17 171530 TURB.html</td>
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<tr>
<td>SN32072 2006-06-18 102309 DO.html</td>
</tr>
<tr>
<td>SN32072 2006-08-14 134200 QuickCal.html</td>
</tr>
<tr>
<td>SN32072 2006-11-25 121530 PH.html</td>
</tr>
<tr>
<td>SN32072 2007-01-11 154322 Cond.html</td>
</tr>
</tbody>
</table>

**HOW OFTEN TO CALIBRATE**

No sensor will remain in calibration forever. The calibration frequency is almost completely determined by the chemical properties of the fluid being monitored, and the accuracy you wish to achieve from the instrument. For example, when used in relatively clean water, in a normal pH range, at a relatively stable temperature, some sensors could remain in calibration for a couple of weeks or longer. On the other hand, in surface water with a high nutrient content and wide temperature fluctuations, the sensors may need to be cleaned and recalibrated every few days. Your own measurement results are the best guide to the need to recalibrate.

When a sensor or instrument is new, we recommend checking the readings often (say, once a day) to get an idea of the stability of the sensor.

Changes in flow also affect readings. Constant flow will increase the accuracy. This can be achieved with the stirring accessory.

The table below may be used as a very general guideline to how long sensors may be expected to remain in calibration under optimum conditions:

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Expected Calibration Period</th>
</tr>
</thead>
<tbody>
<tr>
<td>pH, ORP</td>
<td>1-2 months</td>
</tr>
<tr>
<td>Conductivity</td>
<td>2-3 months</td>
</tr>
<tr>
<td>D.O. (polarographic)</td>
<td>2-4 weeks</td>
</tr>
<tr>
<td>D.O. (optical, RDO)</td>
<td>up to a year if foil is not damaged</td>
</tr>
<tr>
<td>ISEs</td>
<td>1 day</td>
</tr>
</tbody>
</table>

**TIP:** For additional information on calibration schedules, see the Technical Note on Instrument Calibration in the Downloads section at www.in-situ.com.

**HOW TO CHECK IF A SENSOR IS STILL IN CALIBRATION**

Immerse the sensor in a calibration standard of known value and at the same temperature as the original calibration. Compare the sensor reading to the solution value. Some drift is to be expected, but generally the readings should fall within the sensor’s accuracy specification. If readings fall outside the accuracy specification by an amount that is not acceptable for your current application, recalibration is recommended. You will quickly learn by experience how often you need to recalibrate a given sensor based on usage.

**TIP:** Quick Cal solution may be used for a quick check of pH, ORP, and conductivity. Refer to the values printed on the label.
WHEN TO REPLACE A SENSOR

After a certain amount of use even a complete recalibration will not be able to accurately calculate calibration coefficients. The slope will gradually become lower and lower. At this point the sensor should be replaced. Specific slope guidelines for individual sensors are given in the individual parameter sections below.

USING A STIRRER

In-Situ’s stirrer accessory provides continuous sample circulation or agitation, which can improve the performance of water-quality sensors in a number of applications.

Dissolved oxygen (DO) measurements drop in very stagnant water due to depletion of oxygen next to the membrane. A slight perturbation to the system will cause the DO measurements to return to normal. Stirring is recommended if the instrument is anchored to a fixed structure in stagnant conditions—for example, attached to a pier in a calm lake that has no underwater currents. If the wind is blowing and waves are slightly moving the cable, then stirring is probably not necessary.

ISE sensors in close proximity to each other can sometimes create interferences. Constant stirring can enhance the performance of the ISE sensors.

Sample agitation can also help to improve sensor response time when water-quality conditions are subject to change (e.g., in a moving contaminant plume) and can speed up temperature stabilization.

Attaching the Stirrer for Field Use

The stirrer accessory is easily installed on the MP TROLL 9000. See illustration D earlier in this section.

1. Remove the nose cone from the MP TROLL 9500. Leave the restrictor attached to the instrument.

2. Screw the top of the stirrer (propeller end) to the stainless steel restrictor in place of the nose cone.

3. Start the stirrer; see Starting the Stirrer earlier in this section.

The instrument is ready for use in stagnant water.

How can I find the serial number of a water quality sensor—pH for example?

The software can display the sensor serial number. Do this:

1. Select pH in the Navigation tree

2. Look at the information displayed The serial number is displayed in the Information pane on the right side of the screen (or at the bottom on a PDA)

REFERENCES


A Fish Farmer’s Guide to Understanding Water Quality. LaDon Swann, Dept. of Animal Sciences, Illinois-Indiana Sea Grant Program, Purdue University. On the web at AquaNIC (Aquaculture Network Information Center), aquanic.org.


Water on the Web (WOW). University of Minnesota project initially funded by the National Science Foundation. On the web at www.nrri.umn.edu

Water Quality Sensor Pressure Ratings

<table>
<thead>
<tr>
<th>Sensor</th>
<th>Pressure Rating</th>
<th>Usable Depth</th>
</tr>
</thead>
<tbody>
<tr>
<td>pH</td>
<td>300 PSI</td>
<td>210 Meters</td>
</tr>
<tr>
<td>pH/ORP</td>
<td>300 PSI</td>
<td>210 Meters</td>
</tr>
<tr>
<td>Conductivity</td>
<td>350 PSI</td>
<td>246 Meters</td>
</tr>
<tr>
<td>D.O. polarographic*</td>
<td>350 PSI</td>
<td>246 Meters</td>
</tr>
<tr>
<td>Turbidity</td>
<td>350 PSI</td>
<td>246 Meters</td>
</tr>
<tr>
<td>Wiper</td>
<td>350 PSI</td>
<td>246 Meters</td>
</tr>
<tr>
<td>Chloride</td>
<td>100 PSI</td>
<td>70 Meters</td>
</tr>
<tr>
<td>Ammonium</td>
<td>20 PSI</td>
<td>14 Feet</td>
</tr>
<tr>
<td>Nitrate</td>
<td>20 PSI</td>
<td>14 Feet</td>
</tr>
<tr>
<td>RDO</td>
<td>exceeds rating of the TROLL 9500</td>
<td></td>
</tr>
</tbody>
</table>

* Submersion and retrieval at up to 4 feet per second.
WHAT IS pH?

The term pH is derived from “p” meaning power and “H” for the element hydrogen and literally means “power of hydrogen.” pH is defined as the negative logarithm of the hydrogen ion activity (or concentration in moles/liter):

\[ \text{pH} = - \log [H^+] \text{ or } [H^+] = 10^{-\text{pH}} \]

Water (H₂O) dissociates into hydrogen ions (H⁺) and hydroxide ions (OH⁻) in aqueous solution. At 25°C there are 1.0 x 10⁻⁷ moles/L of hydrogen ions and 1.0 x 10⁻⁷ moles/L of hydroxide ions in pure water. Thus the water is neutral (pH = 7) because there are equal amounts of each ion. Addition of a substance with hydrogen or hydroxide ions will shift the balance and cause the water to become either acidic or basic.

The pH scale ranges from 0 (most acidic) to 14 (most basic or least acidic). A change of 1 pH unit corresponds to a tenfold change in hydrogen ion concentration.

WHY MEASURE pH?

A pH value indicates the amount of hydrogen ion that is present in an aqueous environment. The hydrogen ion concentration gives an indication of the acidity of a substance. pH is an important measurement in natural waters because most chemical and biochemical processes are pH dependent. The physiological chemistry of most living organisms can tolerate only small changes in pH and still provide the chemical reactions that sustain life. The solubility of many chemicals is pH dependent. Thus, pH determines their availability to living organisms.

Typical pH values

<table>
<thead>
<tr>
<th>Fluid</th>
<th>pH units</th>
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</thead>
<tbody>
<tr>
<td>Acid rain</td>
<td>&lt; 5</td>
</tr>
<tr>
<td>Distilled water</td>
<td>5.6</td>
</tr>
<tr>
<td>Most natural waters</td>
<td>8</td>
</tr>
<tr>
<td>Safe for freshwater fish</td>
<td>6-9</td>
</tr>
<tr>
<td>Properly chlorinated swimming pool</td>
<td>7.2 - 7.6</td>
</tr>
</tbody>
</table>

Natural waters usually have pH values in the range of 4 to 9. Most natural waters are slightly basic (~ pH 8) because of the presence of carbonates (CO₃²⁻) and bicarbonates (HCO₃⁻). Extremely fresh water can even be slightly acidic (~ pH 6), depending on the concentration of dissolved carbon dioxide (CO₂). The carbon dioxide combines with water to form a small amount of carbonic acid (H₂CO₃) and this process lowers the pH. Nitrogen oxides (NOx) and sulfur dioxide (SO₂) from automobile exhaust and the burning of coal combine with water in the atmosphere to form nitric (HNO₃) and sulfuric acid (H₂SO₄). This falls to the ground as acid rain and accumulates in surface water. Runoff from mining spoils and the decomposition of plant materials can also cause acidic surface water.

pH values below 5 in natural waters are generally considered to be too acidic. Freshwater fish seem to do well in the pH range of 6 to 9. Acidic drinking water is a concern because of its corrosive characteristics to plumbing and appliances. pH also affects the ammonia/ammonium (NH₃/NH₄⁺) equilibrium in water. Even a small amount of ammonia is detrimental to fish while a moderate amount of ammonia is tolerated. At a pH of 6.5 almost all ammonia is in the form of ammonium. However, as the pH becomes slightly basic, ammonium is changed into harmful ammonia. The lethal dose of ammonia for trout is only 0.2 mg/L.

THE pH SENSOR

pH electrodes use a potentiometric method to measure the pH of a solution. The pH sensor consists of a pH-sensitive glass whose voltage is proportional to the hydrogen ion concentration. A second sensor (electrode) serves as a reference, which supplies a constant stable output. Electrical contact is made with the solution using a saturated potassium chloride (KCl) solution. The electrode behavior is described by the Nernst equation:

\[ E_m = E_s + (2.3 \text{ RT/nF}) \log [H^+] \]

where

\[ E_m \text{ is the potential from the pH electrode,} \]
E₀ is related to the potential of the reference electrode, 
R is the Gas Law constant, 
F is Faraday’s constant, 
T is the temperature in Kelvin, 
n is the ionic charge (+1 for Hydrogen), and 
[H⁺] is the hydrogen ion concentration in moles/L.

The MP TROLL 9500 reads the signal from the pH electrode, the reference electrode, and the temperature and then calculates the pH using the Nernst equation.

**SENSOR INSTALLATION**

The MP TROLL 9500 may be shipped with a pH or combination pH/ORP sensor installed. If installation is necessary, unpack and install the sensor in the MP TROLL 9500 as follows.

**A combination pH/ORP sensor will work properly only in port 1. A pH sensor may be installed in port 1 or 3.**

**TIP:** To ensure optimum response for a new or previously stored sensor, rinse off the soaking solution, then soak the sensor in clean water for at least 15 minutes before calibrating.

1. Remove the restrictor or Cal Cup from the front end of the MP TROLL 9500. This allows access to the sensor block shown below.
2. Remove the sensor hydration bottle and set aside for future use.
3. Rinse the sensor in clean water to remove the soaking solution. Soak the sensor in clean water for at least 15 minutes before calibrating.
4. Remove any moisture or dirt from the area around the port where you will install the sensor, then use the sensor removal tool to remove the plug from the port. Retain the plug for future use.
5. Remove any moisture or dirt from the port connector with a clean swab or tissue.
6. Remove the cap from the sensor connector. Check lubrication of the o-rings. If they appear dry, apply a silicone lubricant before installation.
7. Visually align the sensor connector pins with the port connector pins.
8. Press the sensor into the port until you feel it dock with the port connector. When properly inserted a small gap (the width of the sensor removal tool) remains between the instrument body and the widest part of the sensor, for ease of removal.

**CALIBRATION OVERVIEW**

Several options are available for pH calibration.

- **Quick Cal:** Calibrates all basic sensors (pH, ORP, polarographic D.O., conductivity) at the same time with one convenient solution. A one-point calibration in pH 7; default slope, calculated offset.
- **Traditional calibration–1 point:** requires a single solution; results in calculation of sensor slope and offset for one pH range (pH 4-7 or pH 7-10). Choose solutions that bracket the expected pH range.
- **Traditional calibration–2 point:** requires two solutions; results in calculation of sensor slope and offset for one pH range (pH 4-7 or pH 7-10). Choose solutions that bracket the expected pH range.
• 4.0 and 7.0 pH buffer solutions for neutral to acidic conditions
• 7.0 and 10.0 pH buffer solutions for neutral to basic conditions

• Traditional calibration–3 point: requires three solutions; results in calculation of slope and offset for 2 ranges (pH 4-7 and pH 7-10). The correct slope for the pH values being monitored will automatically be applied. A 3-point calibration is useful when the pH range of the environmental fluid is completely unknown.
• Resets the sensor’s factory defaults. No solutions are required.

Nominal vs. Stable

To shorten the calibration time, you have the option to accept the calibration when “Nominal” stability conditions are achieved. If the early value is accepted, the calibration point will be designated “USER SET” in the calibration report. If the calibration report indicates that calibration was performed through to stability then the instrument will operate as intended by In-Situ’s quality standards.

CALIBRATION SOLUTIONS

Calibration solutions certified to N.I.S.T. standards are supplied in the In-Situ pH Calibration Kit: pH 4, pH 7, pH 10 buffer, and deionized water. Catalog No. 0032080. Solutions are also available separately.

**TIP:** Most solutions are usable beyond their stated expiration date, depending on storage conditions; however, the results cannot be guaranteed.

Primary standard buffer salts are available from the National Bureau of Standards, and may be used in situations where extreme accuracy is required. Commercially available pH buffers may be used.

RECOMMENDED CALIBRATION FREQUENCY

Calibration frequency will depend on the nature of the sample and the degree of accuracy required. In clean water samples, the pH sensor should retain its accuracy for 2-6 weeks before requiring recalibration. Recalibrate the sensor—

• after replacing the reference junction and/or the filling solution,
• during routine, scheduled maintenance,
• every 2-6 weeks in the absence of other indications.

**pH QUICK CAL**

The procedure to Quick Cal the pH sensor (a 1-point calibration at pH 7), along with other sensors in the Basic Sensor Set, may be found in Section 3, Getting Started.

To perform a more accurate traditional calibration, follow the procedure below.

**TIP:** The pH calibration procedure is the same for pH sensors and pH/ORP sensors.

TRADITIONAL pH CALIBRATION PROCEDURE

1. With a pH or pH/ORP sensor installed and plugs or sensors in the other ports, rinse the front end of the MP TROLL 9500 in tap water, then again in deionized water. Shake to dry.

For the most accurate results, follow this with a rinse in a portion of the selected calibration solution.

2. Insure the black PVC base is attached to the Cal Cup, and fill the Cal Cup to the fill line with the selected calibration solution.

• Begin with the lowest buffer value when performing a multi-point calibration.
• With a full complement of sensors installed, use the lower line as a guide.
• With 1 or 2 removable sensors installed, fill to the upper line.

3. Insert the front end of the MP TROLL 9500 into the open end of the Cal Cup. Thread the Cal Cup onto the body until seated against the o-ring, then back off slightly to avoid overtightening.

4. Connect the MP TROLL 9500 to a PC and establish a connection in Win-Situ 4 or Pocket-Situ 4. Win-Situ screens are illustrated here. The Pocket-Situ interface is similar, with the Navigation tree at the top of the screen and the Information pane below it.

**RECOMMENDED CALIBRATION ORDER FOR pH AND ORP**

The pH/ORP sensor requires separate calibrations for pH and ORP. A suggested calibration scenario is as follows:

A. First, Quick-Cal ORP (plus, optionally, other installed Basic sensors). For the procedure, see Section 3, Getting Started.

B. Then, perform a 2- or 3-point Traditional pH calibration as described here.
5. Select the MP TROLL 9500 in the Navigation tree.

The software will automatically detect and display the installed sensors. If one or more sensors is installed in the wrong port, an error message will be displayed. Simply remove the sensor and install it in the correct position, then "refresh" the device before continuing.

6. Click to select pH in the Parameters list. The sensor serial number (SN) and recent calibration information is displayed.

7. Select **Calibrate** to launch the pH Calibration Wizard. A screen like this is displayed.

8. Select the number of calibration points for this calibration, and the pH value of the calibration solution for each point. Cal point 1 is the solution the sensor is soaking in now.

9. Select **Next** to continue.

10. In the next screen, select **Run** to begin the stabilization.

   ![Calibration Wizard Screen](image)

   The display will continuously update as readings are taken and compared against the stabilization criteria.

11. If doing a one-point calibration, go to step 14.

For a multi-point calibration, the Wizard returns to the screen shown at step 10 and waits for you to situate the probe in the next calibration solution and click Run.

---

**Indicators during Calibration**

- **Status:**
  - **NOT TESTED** is displayed until you begin the calibration by selecting Run.
  - **UNSTABLE** indicates the sensor response does not meet the criteria for a valid calibration point.
  - **NOMINAL** indicates the sensor deviation meets early stabilization criteria.

The **Accept** button becomes available when nominal stability is achieved. You may accept the early value, or wait for complete stability. If you accept the early value, the calibration point will be designated “USER SET” in the calibration report. (For more on calibration reports, see “Calibration History” in Section 10.)

- **STABLE** is displayed when the readings have stabilized sufficiently to take a valid calibration point. The calibration proceeds automatically to the next screen.

- **Sensor Reading:** The current sensor response in milliVolts.

- **Sensor Deviation:** Change in sensor response between the last two readings. This enables you to follow the progress of the stabilization, but deviation from the previous reading is not necessarily the best indicator of stability as the software is looking at longer-term trends.
12. Remove the Cal Cup, discard the first solution, rinse the Cal Cup and the front end of the instrument, refill the Cal Cup with the second solution, and attach it to the instrument.

**TIP:** The calibration solution may be flushed down the drain with running water, or saved in a separate container and used to rinse the next time you calibrate with the same solution.

13. Select Run to begin the stabilization for the second calibration point. Status indicators and controls are the same as for the first calibration point (step 10).

For a 3-point calibration, repeat steps 12 and 13.

14. The final screen shows the sensor slope and offset calculated during calibration. For a 3-point calibration, 2 sets of coefficients will be shown.

"Pivot pH" is the point at which the slope characteristics change with a 3-point (2-range) calibration. The correct slope for the pH values being monitored will automatically be applied.

15. Select Finish to program the sensor with the newly calculated calibration coefficients.

**TIP:** You can look at the calibration report right after calibrating, or at any time. See "Calibration History" in Section 10 for details.

**Options for storing the sensor:**
- If the instrument will be used in a day or so, leave the sensors installed. Remove the Cal Cup and rinse it and the sensors. Add 50-100 mL of tap water to the Cal Cup. Return the probe to the Cal Cup for transport to the field site.
- For longer storage, see Sensor Care and Handling, below.

**RESETTING DEFAULT COEFFICIENTS**

The sensor’s calibration may be reset back to factory defaults at any time. As the sensor ages, the coefficients calculated during calibration will deviate more and more from the nominal values, which are derived from new sensors. Default coefficients will give reasonable results when the sensor is relatively new.

1. With a pH or combination pH/ORP sensor installed, establish a connection to the instrument in Win-Situ 4 or Pocket-Situ 4.

2. Select pH in the Parameters list and click Calibrate.

3. In the first screen, select Use Nominal Coefficients, then Next.

4. In the final screen, click Finish to send the values to the sensor.

**SENSOR SLOPE AND OFFSET**

The pH calibration curve pivots around pH 7 (0 mV response). The offset calculated by the software when calibrating at pH 7 will typically be between 372-450 mV. If the offset falls much outside these limits, replace the filling solution or the junction (see the following page). The slope should fall between -54 mV/pH and -62 mV/pH. A calculated slope greater than -50 mV/pH or less than -66 mV/pH indicates that the sensor requires maintenance (see the following page).

**UNITS AND CALCULATED MEASUREMENTS**

Readings from pH channel are displayed in pH units. No calculated measurements are available.

**USAGE RECOMMENDATIONS AND CAUTIONS**

- Temperature compensation is provided in the software to account for measurements taken at temperatures different from the calibration temperature. For most accurate results, try to calibrate at the same temperature as the expected sample temperature.

- A small error in pH will occur in basic solutions (>pH 10) that contain high levels of sodium salts (>0.01M) due to sodium interference. Contact In-Situ for more information.
• pH readings in pure water samples (<100µS/cm conductivity—also known as “low ionic strength” samples) require up to 20 minutes after calibration to stabilize and begin producing accurate results. You may wish to condition the sensor after calibration in a low conductivity solution.

**SENSOR CARE AND HANDLING**

**SENSOR REMOVAL**

Position the yoke of the sensor removal tool at the point where the sensor enters the sensor block and pop the sensor out.

**MAINTENANCE/INSPECTION/CLEANING**

If a film develops on the glass electrode, or if the sensing glass or junction should become dehydrated, the sensor response may be sluggish or erratic, or the sensor may fail to calibrate. In these cases, rinse the sensor in 90% isopropyl alcohol, then soak it in storage solution (Catalog No. 0065370) for at least an hour, or overnight if needed. If this does not restore the response, try soaking in 0.1 M HCl solution for 5-10 minutes, followed by a thorough rinse in clean water. If the response has still not improved, replace the filling solution, or possibly the junction.

**Note:** The following maintenance instructions apply to In-Situ’s newest pH sensor (cat. no. 0059510). Older sensors (cat. no. 0032000, now discontinued) are not user-serviceable.

**REPLACING THE FILLING SOLUTION**

Replace the filling solution every five to six months, or when:

- The sensor fails to calibrate with reasonable slope and offset
- Readings drift
- Readings during calibration at pH 7 are outside the range 0±20 mV

1. Unscrew the reference junction as shown.
2. Holding the sensor at an angle, shake out the filling solution.
3. Protect the connector end of the sensor with the soft cap it shipped with, or wrap the sensor in a paper towel to prevent solution from entering the electrical connector.
4. Using the dispenser cap on the filling solution bottle, insert the tube into the **bottom** of the empty reservoir. Squeeze a steady stream of solution into the reservoir until it overflows and no bubbles are observed. Continue to add solution while withdrawing the tube.
5. Screw in the reference junction, and hand-tighten until snug. Some filling solution will overflow. Wipe the excess off the sensor body.
6. Soak the sensor in tap water for at least 15 minutes.
7. Recalibrate the sensor.

- **If necessary,** thoroughly clean the electrical connector to remove filling solution: Using a disposable pipette, fill the connector with isopropyl alcohol (70% to 100%). Shake to dry. Repeat 3 times. Dry overnight. When thoroughly dry, recalibrate.

**REPLACING THE JUNCTION**

Replace the junction when the sensor fails to calibrate, even after replacing the filling solution.

1. Unscrew the reference junction and discard.
2. Replace the solution and screw in a new junction as above.
3. Soak for 15 minutes in tap water, then recalibrate the sensor.

**TIP:** Keep the junction damp at all times to avoid a lengthy rewetting process.

**STORAGE**

**Short-Term Storage (several days)**

Store in the Cal Cup in tap water.

**Long-Term Storage (several weeks)**

Remove the sensor and store it in the electrode storage bottle with 10-20 mL of storage solution (Catalog No. 0065370). Tighten the cap to prevent drying. Prior to use, condition the sensor by rinsing with deionized or tap water and soaking for 15 minutes.

**REFERENCES**


WHAT IS CONDUCTIVITY?

Electrical conductivity measures the ability of a material to carry an electric current. Lakes, rivers, oceans, and underground aquifers are typically good conductors because they contain dissolved salts and minerals. These salts and minerals dissociate in the presence of water to form negatively and positively charged particles called anions and cations. Anions and cations provide a pathway for the transportation of electrical charges throughout the aqueous medium. For the most part, the higher the concentration of dissolved salts and minerals in water, the better the conductor and the higher the electrical conductivity. Deionized/distilled water is a poor conductor because almost all anions and cations are removed during the deionization/distillation process.

WHY MEASURE CONDUCTIVITY?

Changes in the conductivity of a body of water are often used to indicate an environmental event. For example, a drastic increase in the electrical conductivity of an underground fresh water aquifer located near the ocean could indicate the beginning of salt water intrusion. On the other hand, an increase in the electrical conductivity of a small lake that is completely surrounded by farmland may simply be the result of runoff from a recent rain.

HOW IS CONDUCTIVITY MEASURED?

Conductance is the reciprocal of the resistance, in ohms, measured between two opposing electrodes of a 1 cm cube at a specific temperature. The unit 1/ohm or mho was given the name of Siemens (S) for conductance. It is not practical to require all conductance cells to have the dimensions of an exact cube. To enable the comparison of data from experiments with different conductance cells, the conductance is multiplied by the cell constant to give conductivity in Siemens per centimeter (S/cm). Cell constants are determined for each sensor using a standard solution of known conductivity. The cell constant depends on the electrode area and the amount of separation or distance between the electrodes.

Early conductivity measurements were performed using cells with two electrodes. This method required using three conductivity cells with different cell constants in order to span the range of 1 to 100,000 microSiemens per centimeter (µS/cm). Another inconvenience occurred when deposits formed on the electrodes, thus reducing the measured conductivity of the sample.

The modern four-electrode conductivity cell offers many advantages over the two-electrode method. It contains two drive electrodes and two sensing electrodes. The sensing electrodes are positioned in a low current area so that electrode fouling is minimized. An alternating current is used to drive the cell. This reduces errors caused by polarization resulting from the application of a direct current.

THE CONDUCTIVITY SENSORS

Two conductivity sensors are available, optimized for performance in different areas of the conductivity range. Chemically resistant electrodes are used for lower reactivity in high conducting samples (carbon electrodes in the low-range sensor, passivated stainless steel electrodes in the high-range sensor).

The conductivity sensors are shorter than the other water quality sensors in order to distance the conductivity cell from the KCl reference solutions in other sensors.

### Typical Conductivity values

<table>
<thead>
<tr>
<th>Type of Water</th>
<th>Conductivity (µS/cm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ultra-pure distilled</td>
<td>0.05</td>
</tr>
<tr>
<td>Distilled water</td>
<td>1.0</td>
</tr>
<tr>
<td>Drinking water</td>
<td>50 to 300</td>
</tr>
<tr>
<td>Surface water</td>
<td>100 to 10,000</td>
</tr>
<tr>
<td>Sea water</td>
<td>40,000 to 55,000</td>
</tr>
<tr>
<td>Great Salt Lake</td>
<td>158,000</td>
</tr>
<tr>
<td>Type</td>
<td>Operating Range</td>
</tr>
<tr>
<td>-------</td>
<td>-----------------</td>
</tr>
<tr>
<td>Low</td>
<td>3 to 50,000 µS/cm</td>
</tr>
<tr>
<td>High</td>
<td>70 to 200,000 µS/cm</td>
</tr>
</tbody>
</table>

**SENSOR INSTALLATION**

The MP TROLL 9500 may be shipped with a conductivity sensor already installed in port 4, as shown on the drawing below. If installation is necessary, unpack and install the conductivity sensor in port 4 as follows.

**The conductivity sensor will function properly only when installed in port 4.**

1. Remove the restrictor from the front end of the MP TROLL 9500. This allows access to the sensor block shown in the drawing below.
2. If there is a cap on the connector end of the sensor, remove it and set it aside for future use.
3. Remove any moisture or dirt from the area around port 4, then use the sensor removal tool to remove the plug from the port. Retain the plug for future use.
4. Remove any moisture or dirt from the port connector with a clean swab or tissue.
5. Check lubrication of the sensor o-rings.

**TIP:** The sensor o-rings require generous lubrication before installation. New sensors will be lubricated at the factory. If the o-rings appear dry, apply a silicone lubricant before installation.

6. Align the mark on the side of the sensor with the mark on the port.
7. Firmly press the sensor into the port until you feel it dock with the connector at the bottom of the port. When properly inserted a small gap (width of the sensor removal tool) remains between the widest part of the sensor and the instrument body, for ease of removal.

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**CALIBRATION**

**OVERVIEW**

The conductivity calibration calculates the cell constant for the conductivity sensor. A one-point calibration is sufficient. Best results will be obtained if you calibrate with the solution for the range you expect to measure, and at the temperature you expect during field use.

**Nominal vs. Stable**

To shorten the calibration time, you have the option to accept the calibration when “Nominal” stability conditions are achieved. If the early value is accepted, the calibration point will be designated “USER SET” in the calibration report. If the calibration report indicates that calibration was performed through to stability then the instrument will operate as intended by In-Situ’s quality standards.

**CALIBRATION SOLUTIONS (PRIMARY STANDARDS)**

Potassium chloride (KCl) calibration solutions certified to N.I.S.T. standards are supplied in the In-Situ Conductivity Calibration Kits. The value on the bottle indicates the solution’s specific conductance (conductivity at 25°C). Select an appropriate solution for your application from the following:

- Fresh water 147 µS/cm solution
- Fresh to brackish water 1,413 µS/cm solution
- Brackish water 12,890 µS/cm solution
- Sea water 58,670 µS/cm solution

The standard conductivity calibration kit includes a quart each of 147 µS/cm, 1,413 µS/cm, 12,890 µS/cm and deionized water. Catalog No. 0032090. Solutions are available separately, and specialized kits are available for fresh and salt water applications.

**TIP:** Most solutions are usable beyond their stated expiration date, depending on storage conditions; however, the results cannot be guaranteed. The 147 µS/cm solution should be refrigerated.

**Calibrating with Other Solutions**

A custom solution may be used if its specific conductance value (in µS/cm) at 25°C is known. Calibration to secondary standards may also be performed. This involves a manual calculation of the cell constant. See the procedure later in this section.

**RECOMMENDED CALIBRATION FREQUENCY**

Your own experience is the best guide to how often the conductivity sensor will benefit from recalibration. Refer to the general guidelines under “How Often to Calibrate” in Section 10, and to the tech note on “Instrument Calibration.”
CONDUCTIVITY QUICK CAL

The procedure to Quick Cal the conductivity sensor (a 1-point calibration at approximately 8,000 µS/cm), along with other sensors in the Basic Sensor Set, may be found in Section 3, Getting Started. To perform a more accurate calibration for a specific conductivity range, follow the procedure below.

TRADITIONAL CONDUCTIVITY CALIBRATION PROCEDURE

1. With a conductivity sensor installed and plugs or sensors in the other sensor ports, rinse the front end of the MP TROLL 9500.

To calibrate using a medium- to high-range solution, rinse in tap water and shake to dry.

To calibrate using a low-range solution, it is important to rinse well; we recommend a rinse with tap water, then with deionized water, followed by a rinse with the solution to be used for calibration.

TIP: For highest accuracy, conductivity sensors should be wetted for 15-30 minutes immediately prior to calibration. This immersion can be in either clean water or the conductivity calibration solution.

2. Insure the black PVC base is attached to the Cal Cup, and fill the Cal Cup with the selected calibration solution.

• Low-range sensor: Fill to the lower or upper line depending on the number of sensors installed (fewer sensors require more solution).
• High-range sensor: Fill to or above the upper line, depending on the sensor load. You need enough solution to immerse the sensor’s side ports.

3. Insert the front end of the MP TROLL 9500 into the open end of the Cal Cup. Thread the Cal Cup onto the body until seated against the o-ring, then back off slightly to avoid overtightening.

• Low-range sensor: the open area of the sensor should be completely immersed.
• High-range sensor: the side openings should be completely immersed.
• The temperature sensor should be immersed in about an inch of liquid.
• If any air bubbles are visible on the sensor, tap the sides or bottom of the Cal Cup to dislodge them. Or invert the Cal Cup a couple of times.

4. Connect the MP TROLL 9500 to a PC and establish a connection in Win-Situ 4 or Pocket-Situ 4. Win-Situ screens are illustrated here. The Pocket-Situ interface is similar, with the Navigation tree at the top of the screen and the Information pane below it.

5. Select the MP TROLL 9500 in the Navigation tree.

The software will detect and display the installed sensors. If any sensor is installed in the wrong port, an error message will be displayed. Simply remove the sensor and install it in the correct position, then “refresh” the device before continuing.

6. Select conductivity in the Parameters list. The sensor serial number (SN), type, and recent calibration information is shown.

7. Select Calibrate.

The Conductivity Calibration Wizard starts. Available calibration ranges will depend on the sensor installed (high or low range)

8. Select the calibration solution the sensor is soaking in.

For a custom solution, select Other and enter the Specific Conductance of the solution (conductivity corrected to 25°C) in µS/cm.

9. Select Next to continue.
10. In the next screen, select **Run** to begin the stabilization.

   ![Conductivity Calibration Wizard - Sensor Calibration](image)

   The display will continuously update as readings are taken and compared against the stabilization criteria.

   • **Status indicators:**

     - **NOT TESTED** is displayed until you begin the calibration by selecting Run.
     - **UNSTABLE** indicates the sensor response does not meet the criteria for a valid calibration point.
     - **NOMINAL** indicates the sensor deviation meets early stabilization criteria.

     The **Accept** button becomes available when nominal stability is achieved. You may accept the early value, or wait for complete stability. If you accept the early value, the calibration point will be designated “USER SET” in the calibration report. (For more on calibration reports, see “Calibration History” in Section 10.)

     **STABLE** is displayed when the readings have stabilized sufficiently to take a valid calibration point. The calibration proceeds automatically to the next screen.

   • Temperature at the time of calibration is displayed for your information.

   • **Sensor Reading:** The current sensor response in ohms.

   • **Sensor Deviation:** Change in sensor response between the last two readings. This enables you to follow the progress of the stabilization, but deviation from the previous reading is not necessarily the best indicator of stability as the software is looking at longer-term trends.

11. The final screen shows the new cell constant (Kcell) calculated for the selected range during the calibration process.

   ![Conductivity Calibration Wizard - Finish Calibration](image)

   Typical cell constants:
   - Low-range sensor: 0.32 – 0.39
   - High-range sensor: 4.4 – 5.8

   The displayed cell constant may be edited.

12. Select **Finish** to program the sensor with the displayed cell constant.

   **TIP:** You can look at the calibration report right after calibrating, or at any time. See “Calibration History” in Section 10 for details.

   The conductivity sensor is now calibrated and ready to use in the range for which it was calibrated.

   **TIP:** The calibration solution may be flushed down the drain with running water, or saved in a separate container and used to rinse the sensors the next time you calibrate with the same solution.

**Options for storing sensors:**

• If the instrument will be used in a day or so, leave all the sensors installed in the MP TROLL 9500. The conductivity sensor does not require any special storage conditions, but other sensors do. Refer to the relevant sections of this manual for storage recommendations for other installed sensors.

• Remove the conductivity sensor from the MP TROLL 9500, rinse it, and store it dry.
USING A CONDUCTIVITY METER AS A SECONDARY STANDARD

If a conductivity meter is available for comparison, the cell constant (Kcell) for the conductivity sensor can be calculated by hand and entered manually into the software.

1. Note the current cell constant. Immerse the MP TROLL with conductivity sensor in a solution. Take and record the reading.

2. Take and record a reading in the same solution with a conductivity meter.

3. Solve the following for X:

\[
\frac{\text{Current Kcell}}{\text{Reading with this Kcell}} = \frac{X}{\text{Conductivity meter reading}}
\]

4. This is the new cell constant. Enter this value in the software as described below.

ENTERING A CELL CONSTANT MANUALLY

A cell constant may be entered “manually” without running a complete calibration.

1. With a conductivity sensor installed, establish a connection to the instrument in Win-Situ 4 or Pocket-Situ 4.

2. Select Conductivity in the Parameters list and click **Calibrate**.

3. Select Other, and key in any reasonable value. Press **Next** twice to get to the final screen.

4. In the final screen, key in the desired cell constant.

5. Click **Finish** to send the new value to the sensor.

UNITS AND CALCULATED MEASUREMENTS

Basic Unit: AC

Absolute (or “actual”) conductivity, without temperature compensation, is the basic unit for the conductivity sensor. Measurements may be displayed in:

- microSiemens per centimeter (µS/cm AC)
- milliSiemens per centimeter (mS/cm AC)

The following units are also available for displaying derived measurements calculated from the conductivity channel output:

- Specific Conductance (SC)
- Total Dissolved Solids (TDS)
- TDS concentration in mg/L

TIP: Since the specific ionic composition of all analytes cannot be known, the conversions provided in the software are reasonably good estimates of Specific Conductance and Total Dissolved Solids. The resulting derived values should be treated with caution.

Specific Conductance (SC)

Specific Conductance is conductivity corrected to 25°C. The software estimates what the conductivity would be at 25°C to enable comparison between measurements made at different temperatures.

The conversion requires a temperature coefficient for the solution being measured. By convention, the temperature coefficient for potassium chloride (KCl) calibration standards is used. Specific conductance is calculated from:

\[
SC = \frac{AC}{[1 + 0.0191 \times (\text{Temp.} - 25.0)]}
\]

where

- AC is the actual conductivity in µS/cm
- 0.0191 is a nominal temperature coefficient for KCl solutions
- Temp. is the solution temperature in degrees C.

The correction factor of 0.0191 (1.91% /°C) for KCl solutions is a reasonable approximation for samples containing sodium and chloride salts (i.e., seawater). For comparison, the table below lists correction factors for other solution types.

<table>
<thead>
<tr>
<th>Solution</th>
<th>Correction factor (%/°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>5% H₂SO₄</td>
<td>0.96</td>
</tr>
<tr>
<td>10% HCl</td>
<td>1.32</td>
</tr>
<tr>
<td>5% NaOH</td>
<td>1.7</td>
</tr>
<tr>
<td>Dilute NH₃</td>
<td>1.88</td>
</tr>
<tr>
<td>KCl salt (default)</td>
<td>1.91</td>
</tr>
<tr>
<td>NaCl salt</td>
<td>2.12</td>
</tr>
<tr>
<td>98% H₂SO₄</td>
<td>2.84</td>
</tr>
<tr>
<td>Ultrapure water</td>
<td>4.55</td>
</tr>
<tr>
<td>Sugar solution</td>
<td>5.64</td>
</tr>
</tbody>
</table>

Units: microSiemens per centimeter (µS/cm SC)
Total Dissolved Solids (TDS)

A factor of 0.65 x specific conductance is used to estimate Total Dissolved Solids (TDS). This was chosen for general applicability. Remember that ions in solution will vary, and this general conversion factor will not fit all situations exactly. Units: mg/L

Resistivity

The reciprocal of conductance is resistance. Resistivity is the resistance times the cell constant. Resistivity is useful when monitoring pure water. Units: Kohms cm

Salinity

Calculated from conductivity and temperature using the Practical Salinity Scale adjusted for low salinities. Units: PSU (Practical Salinity Units)

The original Practical Salinity Scale (1978) was considered valid for a range of 2-42 PSU; “standard seawater” is defined as having a value of exactly 35. In 1986, there was an adjustment to the scale for better accuracy with low salinities. That adjusted scale is considered valid for a range of 0-40 PSU. Above 2 PSU there is no significant difference between the two scales. “Fresh” water would typically have values below 1, and typically very close to 0.

USAGE RECOMMENDATIONS AND CAUTIONS

CONDUCTIVITY AND TEMPERATURE

Conductivity is a function of temperature. According to the EPA, temperature variations and corrections represent the largest source of potential error in conductivity measurements. A 0.1°C change in temperature can cause a 0.2% change in conductivity.

Specific conductance is the conductivity of a substance at 25°C, and measurements are usually standardized to 25°C when it is necessary to compare data. When the temperature of a sample and its conductivity at that temperature are known, the software can extrapolate the conductivity to 25°C.

SENSOR CARE AND HANDLING

SENSOR REMOVAL

Position the yoke of the sensor removal tool at the point where the sensor enters the sensor block and pry the sensor upward.

MAINTENANCE/INSPECTION/CLEANING

Check the sensor for fouling of the electrodes. If necessary, flush the sensor with water, or swish in a mild detergent solution and rinse with tap water. A swab or soft-bristle brush may be gently used to clean the electrodes. Remember that the electrodes are made of graphite, which is soft and easily damaged.

STORAGE

Store the sensor dry.

REFERENCES


The International Association for Physical Science of the Ocean (IAPSO) Standard Seawater manufactured by Ocean Scientific International is available in North America from Guildline Instruments Inc. in Lake Mary, Florida.
WHAT IS DISSOLVED OXYGEN?

The amount of dissolved oxygen (D.O.) in both natural water and wastewater is a function of several parameters. D.O. is highly dependent on temperature and atmospheric pressure. An increase in temperature causes a decrease in the amount of oxygen that can dissolve in water. On the other hand, higher atmospheric pressures result in higher D.O. values. Salinity is also a factor. Oxygen solubility is greater in freshwater than in saltwater. There are also chemical and biochemical processes that affect D.O.

Most of the dissolved oxygen in water comes from the atmosphere, but oxygen from the photosynthesis of aquatic plants is also a key source. D.O. levels in lakes and other surface water will actually follow a cyclic or diurnal pattern over the course of a day, rising and falling as light intensity changes from dawn to dusk.

WHY MEASURE DISSOLVED OXYGEN?

Most aquatic life requires an average D.O. value greater than 5.0 milligrams dissolved oxygen per liter of water (mg/L) in order to survive. Although the amount of dissolved oxygen in a body of water fluctuates due to natural processes, large deviations from the norm are usually a result of human activity. Changes in D.O. levels are usually the result of a buildup in organic waste. Organic waste can enter surface water from sewage treatment facilities, runoff from agricultural feed lots or domestic areas and from industrial discharge. Organic wastes often contain nitrates and phosphates. Nitrates and phosphates are nutrients for aquatic plants and algae, stimulating overproduction when present in excessive levels. Accelerated growth of blooms increase the number of photosynthesizing plants, which temporarily increases the amount of dissolved oxygen. However submerged aquatic vegetation eventually experience a reduction in sunlight due to increased coverage on the surface. This decrease in sunlight leads to a reduction in photosynthesis and eventual death. Bacterial processes take over and consume even more dissolved oxygen. Fish and other aquatic species die due to lack of dissolved oxygen. This tragic process is known as eutrophication.

Typical D.O. values

<table>
<thead>
<tr>
<th>Condition</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>100% DO, 0°C, 1 atm, 0 ppm Chlorine*</td>
<td>14.6 mg/L</td>
</tr>
<tr>
<td>100% DO, 20°C, 1 atm, 0 ppm Chlorine</td>
<td>9.09 mg/L</td>
</tr>
<tr>
<td>100% DO, 0°C, 0.75 atm, 0 ppm Chlorine</td>
<td>6.77 mg/L</td>
</tr>
<tr>
<td>100% DO, 20°C, 1 atm, 20 ppm Chlorine</td>
<td>7.35 mg/L</td>
</tr>
<tr>
<td>Safe level for most aquatic life</td>
<td>&gt; 5.0 mg/L</td>
</tr>
</tbody>
</table>

*representative of solute concentration
POLAROGRAPHIC MEASUREMENT OF DISSOLVED OXYGEN

THE POLAROGRAPHIC DISSOLVED OXYGEN SENSOR

THEORY OF OPERATION

The sensor is a Clark-type polarographic sensor consisting of two metallic electrodes in contact with an electrolyte and separated from the measurement water by a polymeric membrane. Dissolved oxygen and other gases diffuse through the membrane into the electrolyte. An electric potential is applied to the electrodes, which causes an electrochemical reaction. Oxygen is reduced at the cathode:

\[ \text{O}_2 + 2\text{H}_2\text{O} + 4e^- \rightarrow 4\text{(OH)}^- \]

while silver is oxidized at the anode:

\[ 4\text{Ag} + 4\text{Cl}^- \rightarrow 4\text{AgCl} + 4e^- \]

The resulting current is proportional to the oxygen crossing the membrane. The electric potential is carefully selected so that only the dissolved oxygen is reduced.

The concentration of dissolved oxygen is usually reported in milligrams of oxygen per liter of water (mg/L), but the sensor actually measures the partial pressure of dissolved oxygen. Other gases such as nitrogen, carbon dioxide, and water vapor are also dissolved in the water. The partial pressure of the oxygen is the fraction of the oxygen multiplied by the total pressure of all the gases. This value is also a function of water temperature and water salinity. The maximum amount of oxygen that can be dissolved in water at a given atmospheric pressure, water temperature, and salinity (100% D.O.) can be calculated from first principles. D.O. measurements taken in the field are then compared to the 100% D.O. value. D.O. measurements of surface water are typically less than the 100% D.O. value due to the presence of biological and chemical processes that consume oxygen. Field measurements are corrected for changes in temperature, air pressure, and salinity.

During the electrochemical process dissolved oxygen is consumed while silver chloride (AgCl) is deposited on the anode. In time, both processes will adversely affect the stability and accuracy of the D.O. measurements. Depletion of oxygen near the membrane will cause readings to decrease when measuring D.O. in stagnant water. The use of a stirrer, or similar mechanism to increase water movement, alleviates this problem.

SENSOR CONDITIONING

As soon as the software “recognizes” and displays the D.O. sensor in port 2, powering of the D.O. channel begins. A low-level current is applied continuously to the D.O. circuitry, resulting in continuous polarization. This is similar to laboratory instruments for measuring dissolved oxygen, which are always “on.” A certain amount of conditioning or “warm-up” time is necessary for the sensor to return accurate readings during calibration and use.

Before calibrating a new D.O. sensor, or a sensor with a new membrane, we recommend that you allow a minimum of two hours for conditioning. For stable long-term performance and faster stability during calibration, we recommend 10 hours of conditioning. If the D.O. sensor is installed when you receive the instrument, it will be conditioned and ready to calibrate. If you remove the sensor, be sure to allow for another period of conditioning before you calibrate.

**TIP:** Testing has shown that 10 hours of conditioning yields very stable long-term performance.

SENSOR INSTALLATION

The MP TROLL 9500 may be shipped with a polarographic D.O. sensor installed in port 2. When the sensor is shipped in the instrument, it is pre-conditioned and ready for calibration.

If installation is necessary, unpack, fill, install, and condition a polarographic D.O. sensor as follows.

**FILL THE MEMBRANE MODULE**

New sensors are shipped with a dry membrane module loosely attached.

1. Remove the soft protective caps from the membrane end and the connector end of the sensor.
2. Remove the membrane module from the sensor body and fill with electrolyte as follows: Holding the membrane module open-end up, position the electrolyte dispenser against the side of the module without touching the membrane. Fill slowly.
   **TIP:** To eliminate air bubbles, tap the side of the module briskly with your fingernail.
3. Insert the sensor into the open end of the membrane module. To minimize air, some of the electrolyte should overflow from the open end as the sensor is inserted.
4. Thread the membrane module to the D.O. sensor. 

Be sure the membrane does not leak. You should not see any drops on the surface. There should be no visible air bubbles.

5. Install and condition the sensor as described below. Then you’re ready to calibrate.

**INSTALL THE SENSOR IN THE MP TROLL 9500**

The polarographic D.O. sensor will function properly only when installed in *port 2*.

1. Remove the restrictor or Cal Cup from the front end of the MP TROLL 9500. This allows access to the sensor block shown in the drawing below.

2. Remove any moisture or dirt from the area around port 2, then use the sensor removal tool to remove the plug or sensor from port 2. Retain the plug for future use.

3. Remove any moisture or dirt from the port connector with a clean swab or tissue.

4. Check lubrication of the sensor o-rings.

**TIP:** The sensor o-rings require generous lubrication before installation. New sensors will be lubricated at the factory. If the o-rings appear dry, apply a silicone lubricant before installation.

5. Handling the sensor by the sides, not the tip, align the mark on the side of the sensor with the mark on the port.

Avoid touching the membrane at the tip of the sensor. Contaminants on the membrane can change its properties and affect measurements.

6. Use the sensor insertion tool to press the sensor into the port until you feel it dock with the connector at the bottom. When properly inserted a small gap (width of the sensor removal tool) remains between the widest part of the sensor and the instrument body, for ease of removal.

7. Turn the sensor “on” and condition it, as described next.

**CONDITION A NEWLY INSTALLED SENSOR**

Condition a new sensor, or one with a new membrane, as follows:

1. Fill and install the sensor as described above.

2. Connect the MP TROLL 9500 to a PC and establish a connection in Win-Situ 4 or Pocket-Situ 4.

3. Select the MP TROLL 9500 in the Navigation tree. All installed sensors will be displayed.

   Powering of the D.O. sensor begins as soon as the software recognizes the D.O. sensor and displays it in the Navigation tree. This starts the conditioning process.

   **TIP:** If you plan to calibrate 100% D.O. in air, condition the sensor in a moist environment at ambient pressure—the loosely attached Cal Cup with a small amount of clean water is ideal.

   If you will be calibrating 100% D.O. in water, condition the sensor dry.

   We recommend that you allow the sensor to condition for 2-10 hours before calibrating. Testing has shown that 10 hours of conditioning yields very stable long-term performance.

   **TIP:** It is not necessary to maintain the computer connection; conditioning continues as long as the sensor is installed.

**CALIBRATION OVERVIEW**

Several options are available for calibrating a polarographic sensor.

- **Quick Cal:** Calibrates all Basic sensors (pH, ORP, polarographic D.O., conductivity) at the same time with one convenient solution. This is a 1-point 100% D.O. calibration in air at ambient pressure.

- **Traditional calibration–1 point:** 100% D.O. may be calibrated either in air (saturated with water) at ambient pressure, or in water (saturated with air—for example, using a bubbler). The water method is generally more accurate, as it better represents actual field D.O. measurement conditions.

- **Traditional calibration–2 point:** 100% D.O. may be calibrated either in air or in water. 0% D.O. is calibrated in an oxygen-
depleted solution such as sodium sulfite or nitrogen-saturated water. A 2-point calibration is recommended
• when you expect to measure very low D.O. values (< 5 mg/L),
• when required by Standard Operating Procedures.

Nominal vs. Stable
To shorten the calibration time, you have the option to accept the calibration when “Nominal” stability is achieved. If the early value is accepted, the calibration point will be designated “USER SET” in the calibration report. If the calibration report indicates that calibration was performed through to stability then the instrument will operate as intended by In-Situ’s quality standards.

CALIBRATION SOLUTIONS & EQUIPMENT
100% D.O. calibrations may be performed in water saturated with air, or in air saturated with water. Deionized water is available from In-Situ Inc., but any clean water may be used. In-Situ’s bubbler calibration kit is designed for an efficient water calibration.

An oxygen-depleted solution is used to calibrate the 0% D.O. Sodium sulfite is available from In-Situ Inc. For a cleaner calibration, nitrogen-saturated water may be used.

RECOMMENDED CALIBRATION FREQUENCY
Your own experience is the best guide to how often the polarographic D.O. sensor will benefit from recalibration under conditions of normal usage. Refer to the general guidelines under “How Often to Calibrate” in Section 10, and the tech note on “Instrument Calibration.”

Until a new polarographic sensor has been thoroughly conditioned, it may require more frequent calibration. In the absence of other indications, a calibration should be performed every 2-4 weeks.

In addition, the polarographic D.O. sensor should be conditioned for 2-4 hours and recalibrated in the following circumstances:
• after cleaning the sensor,
• after replacing the membrane module,
• when taking measurements at an elevation different from that at which the sensor was last calibrated.

DISSOLVED OXYGEN CALIBRATION TIPS

Air or Water? The software provides two options for conducting a 100% D.O. calibration:
• in air (saturated with water). This is the condition during a Quick Cal in the inverted Cal Cup with the sensor membrane exposed to air, temperature sensor submerged, and Cal Cup vented to the atmosphere.
• in water (saturated with air). In-Situ’s bubbler calibration kit provides everything needed to create a vigorous bubbling action to insure air-saturated water. The Cal Cup is not used.

Since dissolved oxygen measurements are typically made in water, calibrating in water will often yield better results. When calibrated in air, the membrane’s behavior in water must be estimated.

• The D.O. calibration procedure is very sensitive to changes in temperature. Ideally, it should be done in an area protected from direct sunlight and away from ventilation ducts.
• The nature of the sensor membrane influences the response. Be sure to note the membrane thickness before starting the calibration. (If no thickness is indicated, the membrane is 1-mil Teflon.) Membrane thickness is more important with a 100% calibration in air.

DISSOLVED OXYGEN QUICK CAL

The procedure to Quick Cal the polarographic D.O. sensor, along with other Basic sensors, is in Section 3, Getting Started. This is a single-point 100% calibration in air (saturated with water).

The following traditional calibration is recommended for use when
• the user is required to enter a specific stimulus at the calibration temperature and pressure,
• calibration in water is preferred,
• a 0% calibration point is needed.

TRADITIONAL DISSOLVED OXYGEN CALIBRATION PROCEDURE

You may select a 1-point or a 2-point calibration. The first point (100% saturation) may be taken in air or in water.

Prepare the MP TROLL 9500 for water or air calibration as described in Steps 1-6 on the following page.
Preparation for 100% D.O. Calibration in Water

The bubbler cal kit for the Polarographic D.O. sensor comes with (1) Battery-powered air pump and 2 alkaline D-cells, (2) Bubbler cup with aquarium stone, tubing, check valve and pinch valve, (3) Grey PVC adapter to support the TROLL 9500 in the top of the bubbler cup.

1. Install the batteries in the portable air pump as shown in the diagram on the inside of the lid.

2. Fill the bubbler container almost to the top with clean water. The optimum amount depends on the volume of the sensors installed in the MP TROLL 9500.

   **TIP:** Tap water is fine, unless it is high in salinity. It is easier to achieve 100% oxygen saturation in low-salinity water. Distilled water is readily available and works well.

3. About 10 minutes before calibration, turn on the bubbler. Regulate bubbling with the pinch valve. For best results, run at the full flow rate to achieve saturation; after 10 minutes you can use the control valve to match the water turbulence conditions expected in the field.

4. Before beginning the calibration procedure, note the membrane thickness stamped on the membrane module.

5. With the D.O. sensor installed and plugs or sensors in the other sensor ports, rinse the front end of the MP TROLL 9500 thoroughly in clean water to remove contaminants and traces of fluids used for earlier calibrations.

6. When ready to calibrate, place the adapter securely in the top of the bubbler housing and insert the front (sensor) end of the MP TROLL 9500 into the adapter.
   - Insure the D.O. sensor is not in the aeration path. Rotate as necessary to prevent air bubbles from collecting on the membrane surface.
   - Insure the temperature sensor is submerged at least 0.5” (12 mm) in the aerated water.

   **Allow a few minutes for the temperature to stabilize, especially if you plan to enter a temperature dependent value from a look-up table.**

---

Preparation for 100% D.O. Calibration in Air

**TIP:** Before beginning the calibration procedure, check the side of the membrane module for the membrane thickness.

1. With the D.O. sensor installed and plugs or sensors in the other sensor ports, rinse the front end of the MP TROLL 9500 thoroughly in clean water to remove contaminants and traces of fluids used for earlier calibrations.

2. Dry the D.O. sensor membrane by shaking the probe and/or gently wiping with a soft swab or the corner of a tissue.

3. Rinse the empty Cal Cup and attach it to the MP TROLL 9500. Thread the Cal Cup onto the body until it is seated against the o-ring, then back off slightly to avoid overtightening.

4. Invert the TROLL with Cal Cup attached and remove the black end cap from the Cal Cup.

5. Gently fill the Cal Cup with clean water until the temperature sensor is completely covered and the membrane at the tip of the D.O. sensor is in air. If any water splashes onto the membrane, gently blot the center of the membrane with a clean cotton swab or the corner of a soft lint-free tissue.

6. Loosely attach the end cap to the Cal Cup. For proper venting, a small hole in the threads of the cap should be at least partly visible to achieve ambient pressure conditions.

   You may wish to use a clamp or other support to maintain the TROLL 9500 in this inverted position.

   **Allow a few minutes for the temperature to stabilize, especially if you plan to enter a temperature dependent value from a look-up table.**
7. If not already connected, connect the MP TROLL 9500 to a PC, launch the software, and “find” the device. Win-Situ screens are illustrated here. The Pocket-Situ interface is similar, with the Navigation tree at the top and the Information pane below it.

8. Select the MP TROLL 9500 in the Navigation tree.

The software will automatically detect and display the installed sensors.

9. Select Dissolved Oxygen in the Parameters list.

The sensor serial number (SN) and recent calibration information is displayed, as shown in the screen below.

10. Select Calibrate.

11. Before the DO Calibration Wizard starts, you will be asked how you want to handle barometric pressure. See the box below.

Barometric pressure is important in converting measurement of D.O. concentration to percent saturation, and a value is required for accurate calibration. If the TROLL 9500 cable is vented, an accurate barometric pressure value can be read from the onboard barometric pressure sensor. If the TROLL cable is non-vented, then a barometric pressure value should be entered manually.

After dealing with barometric pressure the D.O. Calibration Wizard displays a screen like the one below:

12. Select the number of calibration points.
   
   1 point—100% D.O.
   2 points—100% and 0% D.O.

13. Select the membrane type (stamped on membrane module, if not, it’s 1-mil Teflon). If calibrating in water, the membrane thickness is not crucial. When calibrating in air, be sure the correct membrane type is selected.

14. Select Air or Water as the medium for the first calibration point.

15. Select the stimulus at saturation:
   
   • Default—This value is calculated by the software at the current temperature and barometric pressure.
   • User Set—Enter a value from a look-up table if required.

Do one of the following:

• If the TROLL 9500 is on vented cable now and will take measurements using vented cable, click No — and you may want to check the “Don’t ask me this again” box.
• If the device is on vented cable now but will take measurements using non-vented cable, click Yes. In the Edit Barometric Channel screen, check the box indicating non-vented cable for measurements but vented cable for calibration/programming.
• If the device is on non-vented cable now and will take measurements on non-vented cable, click Yes. In the Edit Barometric Channel screen, check the box indicating non-vented cable for measurements and enter a barometric pressure value. For help in supplying information if the cable is not vented, see Section 9, Monitoring Barometric Pressure.
16. Click **Next** to continue. A screen like this will be displayed:

![Screen display of calibration wizard](image)

17. Select **Run** to begin stabilization for the first calibration point.

The display will continuously update as readings are taken and compared against the stabilization criteria.

- **Status indicators:**
  - **NOT TESTED** is displayed until you begin the calibration by selecting **Run**.
  - **UNSTABLE** indicates the sensor response does not meet the criteria for a valid calibration point.
  - **NOMINAL** indicates the sensor deviation meets early stabilization criteria.

  The **Accept** button becomes available when nominal stability is achieved. You may accept the early value, or wait for complete stability. If you accept the early value, the status will be designated “USER SET” in the calibration report. (For more on calibration reports, see “Calibration History” in Section 10.)

- **Sensor Reading:** The current sensor response in nanoAmps.
- **Sensor Deviation:** Change in response between the last 2 readings. This enables you to follow the progress of the stabilization, but the deviation from the previous reading is not necessarily the best indicator of stability as the software is looking at longer-term trends.

- **Current temperature and barometric pressure are shown; these values are used to calculate the stimulus.**

  **TIP:** If the calibration stimulus was entered from a look-up table at step 15 and the temperature is not as expected, you may select **Stop**, then **Back** to retrieve the Setup page and re-enter the User Set stimulus.

18. When readings have stabilized (or you click to **Accept** the Nominal result), the calibration will advance automatically.

If doing a 1-point calibration, go to step 20.

For a 2-point calibration, the Wizard displays a screen similar to the one shown below and waits for you to situate the sensor in oxygen-depleted medium—either the Cal Cup filled with sodium sulfite solution, or a nitrogen-saturated water bath. This time, the D.O. sensor membrane should be completely immersed in solution, as well as the temperature sensor.

![Screen display of calibration wizard](image)

Allow about 15 minutes for the sensor to stabilize in the medium.

19. Select **Run** to begin the stabilization for the 0% calibration point.

Controls and status indicators are the same as for the first calibration point (step 17).

**In true 0% conditions, the sensor reading will be 10 nA or less. If the sensor needs maintenance or there is oxygen in the medium, readings will be higher than 10 nA and complete stability will never be reached. If an accurate 0% calibration is important to your application, do not accept Nominal. Cancel the calibration, perform sensor maintenance and/or check the conditions, and repeat the calibration.**
20. When readings have stabilized (or you Accept the Nominal value), the final screen is displayed. The calculated sensor slope and offset are shown.

![Sensor Calibration Screen](image)

21. Select Finish to program the sensor with the new calibration coefficients. The values will be written to the sensor and you will be asked if you want to see the calibration report.

**TIP:** You can look at the calibration report right after calibrating, or at any time. See “Calibration History” in Section 10 for details.

Rinse the sensor and front end of the instrument very thoroughly after calibrating in sodium sulfite solution. A good way to do this is to fill the Cal Cup with water, attach to instrument, and shake vigorously. This may need to be done a couple of times.

**TIP:** The calibration solution may be flushed down the drain with running water, or saved in a separate container and used to rinse the sensors the next time you calibrate with the same solution.

After everything is thoroughly rinsed, the D.O. sensor is ready se.

### Options for storing the sensor:

We recommend you leave the D.O. sensor installed in the instrument; this will assure you are able to take fast D.O. measurements on demand. A little water (distilled, deionized, or tap) or a damp sponge in the Cal Cup will keep the sensor membrane moist.

Remember that, as long as the D.O. sensor is installed, it is being conditioned.

If the sensor is removed from the instrument and then re-installed, conditioning will begin as soon as the sensor is detected by the instrument and displayed in the software.

### SENSOR SLOPE AND OFFSET

The slope of a properly functioning sensor as calculated during the calibration process will typically be in the following ranges, depending on the membrane thickness:

- 1-mil membrane: between 30 and 67 nA/(mg/L)
- 2-mil membrane: between 15 and 34 nA/(mg/L)

If the calculated slope is much outside the stated range, the sensor may need maintenance. Refer to “Sensor Care and Handling” below.

The default offset for Quick Cal and 1-point calibrations is 2 nA. Offsets for 2-point calibrations should fall under 10 nA.

### UNITS AND CALCULATED MEASUREMENTS

Four units are available for dissolved oxygen:

**TIP:** To change unit preferences: In Win-Situ, select Preferences on the Options menu. In Pocket-Situ, select the Home site, then tap Setup in the command bar.

- Oxygen concentration in milligrams of oxygen per liter of water (mg/L). Since a liter of pure water weighs 1000 grams, and a milligram is 1/1000 of a gram, this is equivalent to ppm (parts per million).
- Oxygen concentration in micrograms of oxygen per liter of water (µg/L).
- Oxygen concentration in microMolar (µmol/L), = mg/L x 31.25
- Oxygen saturation in % —100% D.O. being the maximum amount of oxygen that can be dissolved in water at a given atmospheric pressure, water temperature, and salinity. % saturation output is automatically corrected using the TROLL 9500’s temperature, conductivity, and barometric pressure values (from a baro sensor on vented cable or from a user-entered input). If no conductivity sensor is present, salinity is assumed to be zero.

### Why does it take so long for the D.O. 100% to stabilize in air?

Paying attention to several factors can assure the shortest stabilization times possible within the stability algorithm:

- The sensor membrane is perfectly dry.
- The ambient temperature is stable.
- The temperature sensor is submerged.
- The sensor has been fully conditioned.

If these conditions are met, the sensor response should stabilize in 6-15 minutes.

### Section 13: Dissolved Oxygen—Polarographic

Why does it take so long for the D.O. 100% to stabilize in air?

Paying attention to several factors can assure the shortest stabilization times possible within the stability algorithm:

- The sensor membrane is perfectly dry.
- The ambient temperature is stable.
- The temperature sensor is submerged.
- The sensor has been fully conditioned.

If these conditions are met, the sensor response should stabilize in 6-15 minutes.
USAGE RECOMMENDATIONS AND CAUTIONS

The amount of oxygen that can be dissolved in water decreases at higher temperatures and decreases with increasing altitude (i.e., as barometric pressure drops) and salinity. In other words, as water becomes warmer and saltier, it can hold less and less dissolved oxygen.

During tests that include the D.O. channel, the MP TROLL 9500 automatically measures barometric pressure and temperature for compensation of D.O. readings.

The barometric pressure value at the time of calibration is stored in the sensor and will be used to correct D.O. readings for weather-induced pressure fluctuations taken at the same relative barometric pressure as the calibration. However, the large changes in barometric pressure due to changes in elevation are best accommodated by performing a recalibration. If you calibrate at sea level, for example, and use the instrument in the mountains, you should perform a recalibration at the new altitude.

Be sure to supply a barometric pressure value to correctly calculate dissolved oxygen measurements if the sensor will be attached to suspension wire or non-vented cable. This can be done during calibration or prior to setting up a test.

To assure you are able to take fast D.O. measurements on demand, it is best to leave the D.O. sensor installed in the MP TROLL. This takes full advantage of the conditioning and polarization that was accomplished during calibration. After replacing the TROLL 9500 batteries, be sure to power the sensor for an hour or two before use, especially if the batteries were out for a while.

During the first day, some drift is to be expected. After 24 hours or so, the D.O. values can be expected to stabilize.

The D.O. sensor, like the other water-quality sensors, has been tested to 350 psi pressure (246 m, 807 ft). We recommend gradual submersion and retrieval—no faster than 4 ft per second.

STIRRING

Polarographic D.O. measurements drop in very stagnant water due to depletion of oxygen next to the membrane. A slight perturbation to the system will cause the D.O. measurements to return to normal.

Stirring is not necessary for a hand-held instrument, an instrument attached to a boat or floating object, or in any other situation where the water is moving.

Stirring is recommended if the instrument is anchored to a fixed structure in stagnant conditions—for example, attached to a pier in a calm lake that has no underwater currents. If the wind is blowing and waves are slightly moving the cable, then stirring is probably not necessary.

Attaching the Stirrer

Install the stirrer accessory on the MP TROLL 9000 as follows:

1. Remove the nose cone from the MP TROLL 9500 and set it aside.
2. Screw the top of the stirrer (propeller end) to the stainless steel restrictor in place of the nose cone.

Starting the Stirrer

The stirrer is powered by two alkaline D-cells (installed). To start the motor, tighten the end cap.

The magnetic stir bar in the propeller compartment will start to spin. The stir bar is protected by a guard plate that may be removed for cleaning if necessary.

TIP: If the stir bar does not start spinning, try giving it a gentle nudge by sliding a narrow tool such as a screwdriver or key between the protective bars of the guard plate.

To turn the stirrer off, back off the end cap until the stir bar stops spinning.
SENSOR CARE AND HANDLING

The D.O. sensor kit includes the following items for routine maintenance of the D.O. sensor:

- extra membrane module
- electrode filling solution (KCl electrolyte)
- cleaning solution and brush
- polishing strips (for cathode)
- storage bottle
- o-ring lubricant

SENSOR REMOVAL

Position the yoke of the sensor removal tool at the point where the sensor meets the sensor block and pry the sensor upward.

Always handle the sensor by the sides. Avoid touching the membrane at the sensor tip.

MAINTENANCE/INSPECTION

Inspect the sensor and membrane if readings begin to drift.

- Check for discoloration of the electrodes due to silver chloride (AgCl) deposition.
- Inspect the membrane for integrity of the surface, for the presence of algal growth or other contaminants, for crystallization that may indicate a leak in the membrane, and to ensure no air bubbles are trapped under the membrane.

CLEANING THE ELECTRODES

Remove the membrane module and clean the electrodes as follows:

Cathode. Use a polishing strip to buff the platinum cathode until it is shiny. This removes any deposits, increasing the chemically active surface of the electrode for a stronger D.O. signal.

Anode. If the sensor appears to be excessively discolored from its original matte grey color, clean the anode with ammonia and a soft brush. Extreme discoloration may be removed by soaking for a half-hour in ammonia before cleaning with a brush. The surface of the anode should appear uniform, but not necessarily mirror-like.

Regular cleaning will prevent pitting of the anode surface, caused by accumulated silver chloride deposition. Severe pitting cannot be removed; the sole remedy is to replace the sensor.

After cleaning, rinse thoroughly and shake to dry. Then fill and attach a new membrane module as follows.

REPLACING THE MEMBRANE MODULE

The D.O. sensor performs best in clean water. In environments with high organic content, the membrane can become fouled. Rips, tears and other damage will also affect membrane performance. For best results, replace the membrane when the slope and offset calculated during calibration change dramatically.

The current applied is so small that the electrolyte solution can be expected to last longer than the membrane in most applications.

To replace a membrane module:

1. Make sure the area around port 2 is free of dirt and moisture, then remove the sensor. Remove and discard the used membrane module.
2. Inspect and clean the sensor as needed (see above).
3. Fill a new membrane cap with electrolyte and attach it to the sensor. Refer to “Fill the Membrane Module” earlier in this section.
4. Install and condition the sensor. Refer to “Condition a Newly Installed Sensor” earlier in this section.

Remember to condition the sensor for at least 2 hours, preferably 10 hours, before recalibrating with a new membrane. Even with all visible air bubbles removed, a certain amount of gas will be trapped under the membrane. The conditioning period will remove this excess oxygen.

SENSOR LIFE

The sensor body may be expected to last indefinitely so long as the silver coating is not rubbed off the anode during cleaning. The membrane module should be inspected regularly and replaced when it shows wear or damage and when the slope and offset calculated during calibration change dramatically.

STORAGE

Short-Term Storage (up to a two weeks)

Store assembled with membrane immersed in water. A suitable storage bottle is included in the sensor box.
Long-Term Storage

Remove the membrane module, rinse with deionized water, cap and store dry.

REFERENCES


OPTICAL MEASUREMENT OF DISSOLVED OXYGEN

THE RDO OPTICAL DISSOLVED OXYGEN SENSOR

The In-Situ RDO® optical dissolved oxygen sensor measures dissolved oxygen using the principle of “dynamic luminescence quenching.” Certain molecules, called “lumiphores,” fluoresce when excited by light of a specific wavelength. Oxygen molecules act to quench this fluorescence. The lumiphores in the sensor are embedded in a gas-permeable sensing foil in a replaceable cap.

The sensor optics include a lens, blue LED and filter, red LED and filter, and a photodetector. When the blue LED emits light, the sensing foil emits red photons; the presence of oxygen in the foil causes a reduction in red light detected by the photodiode. The phase difference between the blue excitation light and the returned red light is measured, and the result is used to compute dissolved oxygen.

This method measures the “phase” (or delay) of the returned signal, and is thus based on the “lifetime” rather than the “intensity” of the luminescence.

COMPARISON TO POLAROGRAPHIC D.O. SENSOR

The RDO optical dissolved oxygen sensor offers several advantages over the more traditional electrochemical cell. Its solid-state design does not use membranes, filling solution, or other consumables. The sensor does not consume oxygen, thus it does not require flow past the sensor for measurement of DO. It does not require conditioning before use. It exhibits very little drift, therefore it does not require frequent calibration. In the absence of biofouling, it does not need frequent routine maintenance.

The RDO Sensor and Salinity

Unlike an electrochemical cell, the RDO sensor does not respond to changes in salinity. Since the absolute solubility of oxygen is lower in saline water, it is advantageous to compensate DO concentration readings (µg/L, mg/L, µmol/L) to ensure that the sensor accurately reports the concentration of dissolved oxygen in the presence of significant salinity. This can be accomplished by storing a salinity value in the sensor before taking measurements. The compensation algorithm is applied internally before concentration is reported. The degree of compensation is minimal at very low salinities, and several percent of reading at oceanic salinity levels.

The software prompts for a salinity value when you add a test. The value can be changed at any time by selecting the RDO sensor in the Navigation tree and clicking Edit, then “Edit RDO Salinity Value.”

SENSOR INSTALLATION

You will need—

- TROLL 9500 water quality instrument
- Alkaline batteries

Alternatively use Saft LSH-20 3.6V lithium D cells. Use of any other lithium battery will void the warranty of the RDO sensor and the TROLL 9500.

- Latest version of TROLL 9500 firmware—this comes loaded in a new instrument, or is available with the download of new software at www.in-situ.com
- RDO sensor package, with
- Latest version of Win-Situ 4/ Pocket-Situ 4 software—this is shipped on a CD with a new TROLL 9500 instrument, or is available at www.in-situ.com.
- TROLL Com communication cable
- Desktop/laptop PC or RuggedReader® handheld PC

UNPACK THE RDO CABLE CONNECT SENSOR

The RDO cable connect sensor arrives installed in a 3-part adapter/restrictor/nose cone assembly. Remove the black restrictor and nose
The soft Cal Cup insert enables calibration of the basic sensors when the RDO sensor is installed. It contains:

- An extra screw for the adapter plate
- A sponge to create a moist environment for temporary storage of installed sensors that need to be kept wet

UNPACK THE RDO DIRECT CONNECT SENSOR

The RDO direct connect sensor ships in a box as shown below:

INSTALLING THE RDO SENSOR CAP

1. Remove the red protective dust cap from the sensor and save it for later use.
2. Use the supplied lens wipe to clean the lens of the sensor, if necessary.
3. Remove the RDO sensor cap from its shipping sleeve.
4. Align the arrow on the cap with the index mark on the sensor and firmly press the cap onto the sensor, without twisting, until it seals over the probe body.
   - Keep the cap in its sealed packaging until ready to install it.
   - Make sure that the o-ring is not pinched or rolled inside the cap.
   - The cap’s lifetime is 1 year after the first reading has been taken. Install by the date printed on the packaging.

INSTALLING THE RDO SENSOR

1. If necessary, install batteries in the TROLL 9500 instrument.
   - Please exercise great care in handling, installing, and shipping lithium batteries. Refer to the instructions, cautions and MSDS packed with the batteries.
2. Remove the standard restrictor or Cal Cup from the TROLL 9500 instrument (if attached). This allows access to the sensor block.
3. Determine the installation port(s) for the RDO sensor. For the cable connect, choose port 1 or 3. For the direct connect, you must choose ports 2 and 3 (with the connector pins in port 3).
4. Use the Sensor Removal tool to remove the plug or existing sensor from the appropriate port. Port 1 is normally easier to access.
   - TIP: If installing in port 3, you may wish to remove the port 2 sensor temporarily (if installed).
5. Attach the RDO Sensor to the TROLL 9500:
   a. Remove the soft cap from the connector end of the direct connect RDO sensor. Attach the direct connect sensor and any other remaining sensors (in ports 1 and 4), if applicable. Proceed to step 6.
   b. For the cable connect sensor, insert the front end of the TROLL 9500 instrument through the large hole in the adapter, alongside the RDO sensor.
c. Press until the instrument’s sensor block surface is flush with the adapter plate surface. Align the RDO sensor between sensor ports 1 and 2. Align the open hole for the RDO sensor cable beside port 1. Refer to the drawing below.

d. Tighten the adapter screw with a Phillips head screwdriver, not overly tight.

e. Feed the short black cable up through the open hole, so it is on the same side of the adapter plate as the TROLL 9500 sensor block (refer to the drawing below).

f. Remove the soft cap from the connector end of the RDO sensor cable harness. Align the mark on the side of the sensor with the alignment mark on the selected port (1 or 3). Or just visually align the 3-pin connector on the cable harness with the connector in the selected port.

g. Press the cable harness into the port until you feel it dock with the connector. When properly inserted a small gap (width of the sensor removal tool) remains between the instrument body and the widest part of the cable harness, for ease of removal.

Connected to the TROLL 9500, the RDO sensor looks like this:

6. Remove the RDO sensor cap from its shipping/storage sleeve. Align the arrow with the index mark and firmly press the cap onto the sensor, without twisting, until it seals over the probe body. Make sure that the o-rings are not pinched or rolled between the cap and the sensor.

Avoid allowing moisture, including atmospheric humidity, inside the cap. Keep the cap in its sealed packaging until you are ready to install it. Install promptly. Make sure that o-ring grooves are dry and the o-ring is not rolled or pinched inside the cap.

The cap’s lifetime is 1 year after the first reading has been taken with the TROLL 9500 instrument. Install by the date printed on the packaging.

7. Perform a 2-point calibration on the sensor, as described below.

8. Attach the restrictor and nose cone. (If a turbidity sensor or turbidity wiper are installed on the cable connect model, pull the slack in the adapter cable up against the probe so that it does not interfere with wiper movement or turbidity readings.)

9. If using the cable connect sensor, pull the slack in the adapter cable up against the probe so that it does not interfere with wiper movement or turbidity readings.

**CALIBRATION**

**OVERVIEW**

**Nominal vs. Stable**

To shorten the calibration time, you have the option to accept the calibration when “Nominal” stability is achieved. If the early value is accepted, the calibration point will be designated “USER SET” in the calibration report. If the calibration report indicates that calibration was performed through to stability then the instrument will operate as intended by In-Situ Inc.’s quality standards. For more on calibration reports, see Section 10 of this manual.

**TIP:** During calibration, salinity is set to 0 PSU. A salinity value stored in the sensor is restored after calibration is complete. For more information on storing a fixed salinity value in the sensor, see The RDO Sensor and Salinity earlier in this chapter.
CALIBRATION SOLUTIONS

100% DO calibration is performed in water saturated with air. For best results, use the In-Situ RDO Bubbler Cal kit (Cat. No. 0048580).

0% DO calibration is performed in an oxygen-depleted solution. Sodium sulfite (Na₂SO₃, Catalog No. 0017670) is available from In-Situ Inc. Bubbled nitrogen may be used; in this case allow plenty of time for the oxygen to be completely purged from the water.

RECOMMENDED CALIBRATION FREQUENCY

Calibration frequency is more predictable than with electrochemical DO sensors, since the sensor does not drift appreciably. If the foil is not mechanically damaged or removed, calibration can last three months or more.

**TIP:** For best results, check the RDO sensor several times per year in 100% air-saturated water.

TRADITIONAL 2-POINT CALIBRATION

Ideally, the RDO sensor should be calibrated under stable and controlled conditions, like those found in a laboratory setting. However, field calibration can also be performed. Calibration solutions should be close in temperature to the sample matrix.

One or two calibration points can be taken. If only one point is taken, the software uses the results of a previous calibration for the other point.

A 2-point calibration (100% and 0%) is recommended
- When you expect to measure very low DO values (< 5 mg/L),
- When required by Standard Operating Procedures,
- After replacing the sensor cap

OXYGEN SATURATION POINT

1. Submerge the RDO sensor in a container of clean water aerated with the In-Situ bubbler. Ensure that the sensor is completely submerged and the sensor cap is not directly in the bubble stream.

Allow 5-10 minutes for temperature and oxygen equilibration.

**TIP:** The equilibration time depends on the condition of the water with respect to temperature and exposure to air.

2. Connect the TROLL 9500 to a PC and establish a connection in Win-Situ 4 or Pocket-Situ 4. Win-Situ screens are illustrated here. The Pocket-Situ interface is similar, with the Navigation tree at the top and the Information pane below it.

3. Select the TROLL 9500 in the Navigation tree. The software will automatically detect and display the installed sensors.

4. Select Rugged Dissolved Oxygen in the Parameters list.

Information on the RDO sensor is shown, including its serial number (SN), the foil batch number, and recent calibration information, as shown in the large screen at the bottom of the page.

5. Select Calibrate.

6. Before the RDO Calibration Wizard starts, you will be asked how you want to handle barometric pressure. See the sidebar on page 93.
10. When readings have stabilized, the screen will appear similar to the one below, and you have several choices:

- Select **Next** to display the Zero Oxygen Point screen. Continue with step 11 to calibrate at 0%.
- Select **Next**, then **Next** again to finalize the calibration if you are performing a one-point calibration. Go to step 14.
- Select **Run** to run the oxygen saturation point again if you believe the sensor values are incorrect. Go back to step 9.

**TIP:** The warning box below will appear if the slope is out of range. Repeat the 100% saturation calibration point.

**Barometric Pressure Options**

Cable venting is essential to obtain accurate measurements, and the software cannot tell if the cable is vented, so please take a moment to supply this information. Do one of the following:

- If the TROLL 9500 is on vented cable now and will be deployed on non-vented cable, click **No** — and you may want to check the “Don’t ask me this again” box.
- If the device is on vented cable now but will be deployed on non-vented cable, click **Yes**. In the next screen, check the box indicating non-vented cable for deployment but vented cable for calibration/programming.
- If the device is on non-vented cable now and will be deployed on non-vented cable, click **Yes**. In the next screen, check the box indicating non-vented cable for deployment and enter a barometric pressure value.

For help, see Section 9, Monitoring Barometric Pressure.

7. Three calibration options appear. Select the first or second option:

- Calibrate: Starting from values stored in the sensor (user-set or factory defaults) for the 0% and 100% calibration, this allows the user to redo one or both calibration points.
- Restore defaults, then calibrate: Restores factory defaults for both 0% and 100%, then allows the user to redo one or both calibration points.
- Restore defaults, don’t calibrate: Restores factory defaults for both 0% and 100% saturation and closes the Calibration Wizard.

8. Follow the instructions for 100% oxygen calibration.

9. After allowing 5 to 10 minutes for temperature stabilization, click the **Run** button. The display will continuously update as readings are taken and compared against the stabilization criteria.

**ZERO OXYGEN POINT**

11. Immerse the RDO sensor in an oxygen-depleted medium:

- Sodium sulfite (Na$_2$SO$_3$) solution is generally reliable, if somewhat messy. Use a laboratory beaker or In-Situ’s special zero-point cal cup. Be sure the small well in front of the sensing foil is filled with solution.
- Nitrogen bubbling requires considerable time for oxygen to be completely purged from the water.
- Submerge the sensor completely, and check to be sure that there are no air bubbles on the sensing foil.
- Allow up to 15 minutes for temperature equilibration.
12. When you are ready to take the calibration point, click or tap Run in the Zero Oxygen Point screen.

The display will continuously update as readings are taken and compared against the stabilization criteria. Controls and status indicators are the same as for the 100% calibration point.

13. When readings have stabilized, the screen will appear similar to the one shown in step 10, and you have two choices:

- Select Next to finalize the calibration. Continue with step 14.
- Select Back to calibrate the Oxygen Saturation point (step 9).

**TIP:** Sodium sulfite consumes oxygen aggressively. If performing the oxygen saturation point next, be sure to rinse the TROLL 9500 and RDO sensor thoroughly to avoid cross-contamination.

- Select Run to run the oxygen saturation point again if you believe the sensor values are incorrect. Go back to step 12.

**TIP:** A warning box will appear if the slope is out of range (normal range is 0.9 to 1.1 with an offset of ±0.1). Repeat the zero point.

**FINALIZE THE CALIBRATION**

14. The final calibration screen for a 1-point calibration at saturation was performed. To finalize the calibration, click Finish.

**Status indicators:**

- **NOT TESTED** appears until you begin the calibration by selecting Run.
- **UNSTABLE** indicates the sensor response does not meet the criteria for a valid calibration point.
- **NOMINAL** indicates the deviation meets early stabilization criteria.
  - The Accept button becomes available when nominal stability is achieved. You may accept the early value, or wait for complete stability. If you accept the early value, the status will be designated “USER SET” in the calibration report. (For more on calibration reports and the difference between Nominal and Stable, see Section 10 of this manual.)
- **STABLE** is displayed when the readings have stabilized sufficiently to take a valid calibration point.
  - Reading: The temperature sensor response is shown in degrees C, and RDO sensor response is shown in mg/L. Win-Situ automatically detects stability in both parameters.
  - Deviation: Change in response between the last 2 readings. This enables you to follow the progress of the stabilization, but deviation from the previous reading is not necessarily the best indicator of stability as the software is looking at longer-term trends.
  - Current barometric pressure in your selected unit.

The display will continuously update as readings are taken and compared against the stabilization criteria. Controls and status indicators are the same as for the 100% calibration point. Controls and status indicators are the same as for the 100% calibration point.
TROLL 9500 Operator’s Manual

SECTION 13: DISSOLVED OXYGEN—OPTICAL

The values will be written to the sensor and you will be asked if you want to see the calibration report.

**TIP:** For more on Calibration Reports, see Section 10 of this manual.

The RDO sensor is now calibrated and ready to take readings. To confirm this, take a reading by selecting the RDO sensor in the Navigation tree and clicking the **Read** button.

**TIP:** If the units for the reading are not what you expect, it is easy to change the units selection: In Win-Situ, select Preferences on the Options menu. In Pocket-Situ, select the Home site, then tap Setup in the command bar.

Clean-up

Rinse the sensor and front end of the instrument very thoroughly after calibrating in sodium sulfite solution to avoid cross-contamination. For best results, always use fresh calibration solutions.

**UNITS AND CALCULATED MEASUREMENTS**

Four units are available for dissolved oxygen:

**TIP:** To change unit preferences: In Win-Situ, select Preferences on the Options menu. In Pocket-Situ, select the Home site, then tap Setup in the command bar.

- Oxygen concentration in milligrams of oxygen per liter of water (mg/L). Since a liter of pure water weighs 1000 grams, and a milligram is 1/1000 of a gram, this is equivalent to ppm (parts per million).
- Oxygen concentration in micrograms of oxygen per liter of water (µg/L). This is equivalent to parts per billion (ppb).
- Oxygen concentration in microMolar (µmol/L) = mg/L x 31.25
- Oxygen saturation in % — 100% DO being the maximum amount of oxygen that can be dissolved in water at a given atmospheric pressure, water temperature, and salinity. The percent saturation output is automatically corrected using the TROLL 9500 instrument’s temperature, conductivity, and barometric pressure values (from a baro sensor on vented cable or from a user-entered input). If no conductivity sensor is present, salinity is assumed to be zero.

**USAGE RECOMMENDATIONS AND CAUTIONS**

The amount of oxygen that can be dissolved in water decreases at higher temperatures and with increasing altitude (i.e., as barometric pressure drops) and salinity. In other words, as water becomes warmer and saltier, it can hold less dissolved oxygen.

During tests that include the RDO channel, the TROLL 9500 instrument automatically measures barometric pressure and temperature for compensation of DO readings.

The barometric pressure value at the time of calibration is stored in the sensor and will be used to correct DO readings for weather-induced pressure fluctuations taken at the same relative barometric pressure as the calibration. However, the large changes in barometric pressure due to changes in elevation are best accommodated by performing a recalibration. If you calibrate at sea level, for example, and deploy the instrument in the mountains, you should perform a recalibration at the new altitude.

Exposure to direct sunlight can bleach the sensing foil over time but this effect is minimized with the protection provided by the opaque optical isolation layer. However, the frequent application of strong blue light (blue LED during readings) will eventually have a bleaching effect on the foil. In-Situ recommends sampling intervals of greater than 1 minute for long-term deployments.

**Salinity**

Since the absolute solubility of oxygen is lower in saline waters, it is advantageous to compensate DO concentration readings (µg/L, mg/L, µmol/L) to ensure that the sensor accurately reports the concentration of dissolved oxygen in the presence of significant salinity. This can be accomplished by storing a salinity value in the sensor before taking measurements. The compensation algorithm is applied internally before the concentration is reported. The degree of compensation is minimal at very low salinities, and may affect readings by several percent at higher levels.

To store a salinity value in the RDO sensor, connect in Win-Situ 4 or Pocket-Situ 4, select RDO in the Navigation tree, click Edit and in the next screen select “Edit RDO Salinity Value.”

**SENSOR CARE AND HANDLING**

**BIOFOULING**

Organisms that produce or consume oxygen, if attached to the area of the sensing foil, may artificially influence the sensor’s measurement of the oxygen in the surrounding water. In addition, the sensor’s response time may be increased.

To avoid this the sensor should be cleaned at regular intervals depending on the required accuracy and the fouling condition at the site.
MAINTENANCE AND STORAGE

Cleaning the Sensor Cap
1. Leave the cap on the sensor!
2. Rinse the sensor with clean water from a squirt bottle or spray bottle.
3. Gently wipe with a soft cloth or brush if biofouling is present.
4. If extensive fouling or mineral build-up is present, soak the cap end in vinegar for 15 minutes, then soak in deionized water for 15 minutes.

Do not use organic solvents—they will damage the foil. Do not remove the cap from the sensor prior to brushing.

After cleaning the sensor, perform a 2-point user calibration.

Cleaning the Optical Window (Perform only if changing the cap)
Remove the cap and gently wipe the window with the supplied lens wipe.

Caution: Do not wet the lens area with water or any solution.

Cleaning the Sensor Body
With the sensor cap installed on sensor, gently scrub sensor body with a soft brush. Soak in vinegar and DI water to remove mineral deposits or extensive fouling as in step 4, above.

Storage
Prior to installation: Store in factory supplied container.

Installed: The RDO sensor can be stored wet or dry. When installed with other sensors, it may be useful to store all sensors in a moist environment. In-Situ supplies recyclable plastic calibration and storage sleeves, as well as calibration and storage vessels for this purpose.

Replacing the Sensor Cap
The sensor cap has a 1-year life after the TROLL 9500 instrument takes its first RDO sensor reading. Replacement caps are available from In-Situ Inc. or your authorized In-Situ distributor.
1. Pull the used sensor cap off of the sensor, without twisting.
2. Remove the existing o-rings from the sensor.
3. Use a lint-free cloth to remove any moisture from the sensor body.

Note: Ensure that there is no moisture in the o-ring grooves. Avoid touching or cleaning the lens with anything other than the supplied lens wipe.

RDO Sensor Summary
• When a turbidity wiper is installed, be sure to pull the slack in the RDO adapter cable back against the TROLL 9500 body so it will not interfere with the wiper’s rotation.

• To calibrate the Basic sensors with RDO installed, fill the soft plastic cal insert (it has an orange base) with calibration solution, slide it up around the sensors, including the RDO cable, and use the RDO restrictor as a support during calibration.

• The RDO sensor will work properly only in port 1 or 3 of the TROLL 9500. Remember that a combination pH/ORP sensor will operate properly only in port 1, and a turbidity wiper accessory only in port 3.

• Basic sensors that require hydration (pH and pH/ORP) may be stored in the calibration and storage sleeve for short periods of time. For long-term storage, remove the RDO sensor—it is not necessary to remove it from the adapter—with restrictor and nose cone. Attach the standard TROLL 9500 Cal Cup with a moist sponge in the bottom.

• The RDO sensor and all TROLL 9500 materials are suitable for monitoring in low flow, groundwater, and surface water sites.

4. Use your finger to apply a layer of lubricant around the o-ring grooves. Place the o-rings on the sensor. Apply another thin layer of lubricant to the o-rings and grooves.

Note: Do not transfer grease to the lens or sensor pins.

5. Clean the lens on the sensor with the wipe provided in the kit and allow to dry thoroughly. Inspect for scratches or dirt.

6. Remove the new cap from its sealed packaging and attach it to the sensor, being careful to press firmly, without twisting, until it seals over the probe body. Make sure that the o-rings are not pinched or rolled between the cap and sensor.

7. Perform a 2-point calibration. No conditioning is required prior to use.
WHAT IS ORP?

Oxidation Reduction Potential (ORP) is a measure of a water system's capacity to either release or gain electrons in chemical reactions. The process of oxidation involves losing electrons while reduction involves gaining electrons. Oxidation and reduction (redox) reactions control the behavior of many chemical constituents in drinking water, wastewater, and aquatic environments. The reactivity and solubility of critical elements in living systems is strongly dependent on redox conditions. ORP values are used much like pH values to determine water quality. While pH values characterize the relative state of a system for receiving or donating hydrogen ions (acting as a base or an acid), ORP values characterize the relative state of a system for gaining or losing electrons. ORP values are affected by all oxidizing and reducing agents, not just acids and bases.

WHY MEASURE ORP?

The effect that potable water has on plumbing is directly related to its ORP value. Unfavorable values can cause excessive corrosion, leading to expensive repairs. ORP is one parameter that can be monitored during the disinfecting process for drinking water, swimming pool water, and spa water.

The life expectancy of bacteria in water is related to ORP. In fact, studies have shown that the life span of bacteria in water is more dependent on the ORP value than on the chlorine concentration. For swimming pools at a normal pH value between 7.2 and 7.6, the ORP value must be kept above 700 mV to kill unwanted organisms. Hypochlorite or other oxidizing agents must be added when the ORP falls below 700 mV. In contrast, natural waters need a much lower ORP value in order to support life. Generally ORP values above 400 mV are harmful to aquatic life. Ideally the ORP value in salt water aquariums should be kept between 350 and 390 mV. ORP levels below 300 mV are to be avoided. An oxidizing environment is needed to convert any ammonia (NH₃) to nitrates (NO₃⁻) and nitrates (NO₂⁻). Ammonia levels as low as 0.002 mg/l can be harmful to some fish species.

The determination of ORP is particularly worthwhile in water that contains a relatively high concentration of a redox-active species, e.g., the salts of many metals (Fe²⁺, Fe³⁺) and strong oxidizing (chlorine) and reducing (sulfite ion) agents. Thus, ORP can sometimes be utilized to track the metallic pollution of ground- or surface water, or to determine the chloride content of wastewater effluent. However, ORP is a nonspecific measurement—that is, the measured potential is reflective of a combination of the effects of all the dissolved species in the medium. Because of this factor, the measurement of ORP in relatively clean environmental water (ground, surface, estuarine, and marine) has only limited utility unless a predominant redox-active species is known to be present. Care is required not to "over-interpret" ORP data unless specific information about the site is known.

THE pH/ORP SENSOR

The single-junction, three-electrode sensor uses a potentiometric method to measure pH and ORP in a test solution. The pH electrode consists of a pH-sensitive glass whose voltage is proportional to the hydrogen ion concentration. The ORP electrode serves as an electron donor or acceptor depending upon the test solution. The reference electrode supplies a constant stable output for comparison. Electrical contact is made with the test solution using a saturated potassium chloride (KCl) solution. The electrode behavior is described by the Nernst equation:

\[ E_m = E_o - \frac{(RT)}{nF} \ln \left( \frac{[ox]}{[red]} \right) \]

where

- \( E_{m} \) is the potential from the ORP electrode,
- \( E_o \) is related to the potential of the reference electrode,
- \( R \) is the Gas Law constant,
F is Faraday's constant,  
T is the temperature in Kelvin,  
n is the number of electrons,  
[ox] is the oxidant concentration in moles/L, and  
[red] is the reductant concentration in moles/L.

Most natural waters contain many species that are involved in the redox process so that it is not possible to calculate the ORP using the Nernst equation. All redox species do however reach equilibrium. A Standard solution of known redox potential for a particular ORP electrode is used to calibrate ORP. The sensor then gives a calibrated response in mV when placed in a sample.

**SENSOR INSTALLATION**

The MP TROLL 9500 may be shipped with a pH/ORP sensor installed. If installation is necessary, unpack and install the sensor in port 1 of the MP TROLL 9500 as follows.

1. Remove the restrictor or Cal Cup from the front end of the TROLL 9500. This allows access to the sensor block shown below.
2. Remove the sensor hydration bottle and set aside for future use.
3. Rinse the sensor in clean water to remove the soaking solution. Soak the sensor in clean water for at least 15 minutes before calibrating.
4. Remove any moisture or dirt from the area around port 1, then use the sensor removal tool to remove the plug from the port. Retain the plug for future use.

---

**CALIBRATION OVERVIEW**

A one-point calibration in a solution with a known potential at a given temperature is sufficient to calibrate ORP. Software options:

- **Quick Cal**: Calibrates ORP (and optionally pH, polarographic D.O., and conductivity) at one time with one solution.
- **Traditional calibration**: A one-point calibration in a solution specifically formulated for calibrating ORP. Results in calculation of sensor offset.
- **Default coefficients**: Resets the sensor's factory defaults. No solutions are required.

**Nominal vs. Stable**

To shorten the calibration time, you have the option to accept the calibration when “Nominal” stability conditions are achieved. If the early value is accepted, the calibration point will be designated “USER SET” in the calibration report. If the calibration was performed through to stability (“STABLE” in the calibration report), the sensor will operate as intended by In-Situ’s quality standards.

**CALIBRATION SOLUTIONS**

Only one solution is required for ORP calibration. Quick Cal and ZoBell’s solution are available from In-Situ Inc. A custom solution may be used in a Traditional calibration if its milliVolt value at the calibration temperature is known.

---

Please note that a pH/ORP sensor requires separate calibrations for pH and ORP. The Quick Cal procedure produces excellent results for ORP so long as the solution is stored as recommended and used before its expiration date. After performing a Quick Cal for ORP, we recommend a 2- or 3-point Traditional calibration for pH, as described in Section 11 of this manual.
**RECOMMENDED CALIBRATION ORDER FOR pH AND ORP**

The pH/ORP sensor requires separate calibrations for pH and ORP. A suggested calibration scenario is as follows:

A. First, Quick-Cal ORP (plus, optionally, other installed Basic sensors). For the procedure, see Section 3, Getting Started.

B. Then, perform a 2- or 3-point Traditional pH calibration as described in Section 11 of this manual.

**TRADITIONAL ORP CALIBRATION PROCEDURE**

If you wish to perform a traditional calibration with a dedicated ORP calibration solution, use the following procedure.

1. With a pH/ORP sensor installed and plugs or sensors in the other ports, rinse the front end of the MP TROLL 9500 in clean water. For best results, rinse again in a portion of the calibration solution.

2. Fill the Cal Cup with ZoBell's or other custom ORP calibration solution.

3. Attach the Cal Cup to the MP TROLL 9500. Thread the Cal Cup onto the body until seated against the o-ring, then back off slightly to avoid overtightening.

4. Connect the MP TROLL 9500 to a PC and establish a connection in Win-Situ 4 or Pocket-Situ 4.

5. Select the MP TROLL 9500 in the Navigation tree. The software will automatically detect and display the installed sensors. (This can take a moment.)

6. Select ORP in the Parameters list. The sensor serial number (SN) and recent calibration information is displayed.

7. Select Calibrate to launch the ORP Calibration Wizard.

8. In the ORP Calibration Wizard, select the solution the sensor is soaking in. For ZoBell’s, the reference milliVolt value is available in the software. For a custom solution, select “Other” and enter the mV of the solution at the calibration temperature.

9. Select Next to continue.

10. In the next screen, select Run to begin the stabilization.

   The display will continuously update as readings are taken and compared against the stabilization criteria.

**Indicators during Calibration**

- **Status:**
  - NOT TESTED is displayed until you begin the calibration by selecting Run.
  - UNSTABLE indicates the sensor response does not meet the criteria for a valid calibration point.
  - NOMINAL indicates the sensor deviation meets early stabilization criteria.

The Accept button becomes available when nominal stability is achieved. You may accept the early value, or wait for complete stability. If you accept the early value, the calibration point will be designated “USER SET” in the calibration report. (For more on calibration reports, see “Calibration History” in Section 10.)

- **STABLE** is displayed when the readings have stabilized sufficiently to take a valid calibration point. The calibration proceeds automatically to the next screen.

- **Temperature** is displayed for your information.

- **Sensor Reading:** The current sensor response in milliVolts.

- **Sensor Deviation:** Change in sensor response between the last two readings. This enables you to follow the progress of the stabilization, but deviation from the previous reading is not necessarily the best indicator of stability as the software is looking at longer-term trends.
11. The final screen shows the calculated sensor offset. This value should be 0 mV ± 20 mV.

12. Select **Finish** to write this value to the sensor.

The ORP sensor is now calibrated and ready to use.

**TIP:** You can look at the calibration report right after calibrating, or at any time. See “Calibration History” in Section 10 for details.

**TIP:** The calibration solution may be flushed down the drain with running water, or saved in a separate container and used to rinse the next time you calibrate with the same solution.

**Options for storing sensors:**

- If the instrument will be used in a day or so, leave the sensors installed. Remove the Cal Cup and rinse it and the sensors. Add 50-100 mL of tap water to the Cal Cup. Return the probe to the Cal Cup for transport to the field site.
- For longer storage, see the section on Sensor Care and Handling later in this chapter.

**RESET DEFAULT COEFFICIENTS**

The sensor’s calibration may be reset back to factory defaults at any time. This is a good option if the results of a recent calibration are suspect because the cal solution has been exposed to air or has otherwise deteriorated.

1. With a pH/ORP sensor installed, establish a connection to the instrument in Win-Situ 4 or Pocket-Situ 4.
2. Select ORP in the Parameters list and click **Calibrate.**
3. In the first screen, select Use Default Coefficients, then **Next.**
4. In the final screen, click **Finish** to send the values to the sensor.

**SENSOR OFFSET**

The offset for ORP is a traditional “zero offset,” and is typically 0 ± 20 mV. If the offset calculated during calibration is outside this range, inspect the sensor. If it is clean, then the fault was probably with the cal solution (aging, exposure to air, etc.). In this case, resetting the factory defaults for ORP can often restore the sensor performance.

**UNITS AND CALCULATED MEASUREMENTS**

ORP readings may be displayed in Volts or milliVolts. No calculated measurements are available.

**RECOMMENDED CALIBRATION FREQUENCY**

Calibration frequency will depend on the nature of the sample and the degree of accuracy required. In clean water samples, the sensor could retain its accuracy for 2-6 weeks. Recalibrate the sensor—

- after replacing the reference junction and/or the filling solution,
- during routine, scheduled maintenance,
- every 2-6 weeks in the absence of other indications.

**NORMALIZATION TO STANDARD HYDROGEN ELECTRODE VALUES**

ORP values are dependent on solution composition, temperature, and sensor type. ORP (Eh) values reported in the literature are often normalized to the standard hydrogen electrode as a standard reference electrode. Since the standard hydrogen electrode is extremely fragile, it is impractical to use in the field. In-Situ’s sensor uses a silver/silver-chloride reference electrode in place of the standard hydrogen electrode.

The following equation may be used to normalize the readings reported by our sensor calibrated with our solution to standard hydrogen electrode values (±50 mV).

\[
\text{ORP}_{\text{SHE}} = \text{ORP}_{\text{observed}} + \{215.81 – T_c \ast [0.77942 + T_c \ast 0.001934]\}
\]

where \(\text{ORP}_{\text{SHE}}\) is the sample potential relative to the standard hydrogen electrode, \(\text{ORP}_{\text{observed}}\) is the sample potential relative to the In-Situ reference electrode, and \(T_c\) is the sample temperature in °C.

ORP measured by a sensor immersed in a solution should not be equated with thermodynamic Eh. Differences may occur due to lack of chemical equilibrium, presence of multiple redox couples, sensor poisoning, and other factors.

In addition, like all platinum ORP electrodes, In-Situ’s pH/ORP sensor may give unstable readings in solutions containing chromous, vanadium, titanous, and other ions that are stronger reducing agents than hydrogen or platinum.
**USAGE RECOMMENDATIONS AND CAUTIONS**

- ORP readings vary slightly with temperature, but are not easily corrected because, unlike pH, the ORP value depends on the activity of many ions in solution. pH values are more easily corrected because they are due to the activity of one ion, H⁺.

**SENSOR CARE AND HANDLING**

**SENSOR REMOVAL**

Use the sensor removal tool to pop the sensor out.

**MAINTENANCE/INSPECTION/CLEANING**

If the platinum ORP sensor appears dull or fouled, it may be cleaned with a swab dipped in alcohol. Rub gently until the platinum appears shiny. Rinse in clean water.

If a film develops on the glass electrode, or if the sensing glass or junction become dehydrated, the response may be sluggish or erratic, or the sensor may fail to calibrate. In these cases, rinse the sensor in 90% isopropyl alcohol, then soak in storage solution (Catalog No. 0065370) for at least an hour, overnight if needed. If this does not restore the response, try soaking in 0.1 M HCl solution for 5-10 minutes, followed by a thorough rinse in clean water. If the response has still not improved, replace the filling solution, or the junction.

**Note:** The following maintenance instructions apply to In-Situ's newest pH/ORP sensor (cat. no. 0059520). Older sensors (cat. no. 0032010 & 0032020, now discontinued) are not user-serviceable.

**REPLACING THE FILLING SOLUTION**

Replace the filling solution every 5-6 months, or when:

- The sensor fails to calibrate with reasonable slope and offset
- Readings drift
- Readings during calibration at pH 7 are outside the range 0±20 mV

1. Unscrew the reference junction as shown.

2. Holding the sensor at an angle, shake out the old filling solution.

3. Protect the connector end of the sensor with the soft cap it shipped with, or wrap the sensor in a paper towel to prevent solution from entering the connector.

4. Using the dispenser cap on the filling solution bottle, insert the tube into the bottom of the empty reservoir. Squeeze a steady stream of solution into the reservoir until it overflows and no bubbles are observed.

   Continue to add solution while withdrawing the tube.

5. Screw in the reference junction, and hand-tighten until snug. Some filling solution will overflow. Wipe the excess off the sensor body.

6. Soak the sensor in tap water for at least 15 minutes.

7. Recalibrate the sensor.

   ![If necessary, thoroughly clean the electrical connector to remove filling solution. Using a disposable pipette, fill the connector with isopropyl alcohol (70% to 100%). Shake to dry. Repeat 3 times. Dry overnight. When thoroughly dry, recalibrate.]

**REPLACING THE JUNCTION**

Replace the junction when the sensor fails to calibrate, even after replacing the filling solution.

1. Unscrew the reference junction and discard.

2. Replace the filling solution and screw in a new reference junction as described above.

3. Soak for 15 minutes in tap water, then recalibrate the sensor.

   ![TIP: Keep the junction damp at all times to avoid a lengthy rewetting process.]

**STORAGE**

**Short-Term Storage (several days)**

Store in the Cal Cup in tap water.

**Long-Term Storage (up to several weeks)**

Remove the sensor and store in the electrode storage bottle with 10-20 mL of storage solution (Catalog No. 0065370). Tighten the cap to prevent drying. Prior to use, condition the sensor by rinsing with deionized or tap water and soaking for 15 minutes.

**REFERENCES**

WHAT IS AMMONIUM?

Ammonium (NH₄⁺) is the ionized form of ammonia (NH₃). Ammonia and ammonium are naturally occurring forms of nitrogen, part of the nitrogen cycle. In natural waters they exist in two forms: Dissolved ammonia gas (NH₃) is highly toxic to aquatic life, while ammonium, the ionized form (NH₄⁺), is not. Both may be grouped together as “total ammonia.”

The ammonia/ammonium equilibrium in water is closely related to pH levels. At a pH of 6.5 almost all ammonia is in the form of ammonium. However, as the pH increases (becomes more basic), ammonium is changed into harmful ammonia. Ammonium ions are the predominant species in most unpolluted natural water systems where the pH is typically less than 9. Even a small amount of ammonia is detrimental to fish while a moderate amount of ammonium is tolerated. The lethal dose of ammonia for trout is only 0.2 mg/L.

WHY MEASURE AMMONIUM?

Major sources of ammonium are wastewater from sewage treatment plants, and nitrogen in fertilizers which is transformed to ammonium in soil by microorganisms. Ammonia/ammonium can be a key parameter in the assessment of water and wastewater quality. Measurement of ammonium can yield information on the composition and movement of pollutants in groundwater and surface water, landfill leachate, runoff from agricultural activities, waste concentrations in fisheries, and nutrient levels in natural water bodies.

THE AMMONIUM SENSOR

The In-Situ sensor is an ion-selective electrode (ISE) that is selective for the ammonium ion (NH₄⁺). It is a double-junction combination ISE with a silver/silver-chloride reference half-cell, PVC sensing membrane, and reference electrolyte gel. It measures the concentration in parts per million of ammonium ion in solution (calculated as nitrogen, ppm as N).

SENSOR PREPARATION

To ensure optimum membrane response, the ammonium sensor should be thoroughly hydrated in an appropriate solution before calibration. A good way to do this is to allow the sensor to soak in the solution you plan to use for the first calibration point (lowest concentration) for at least 15 minutes and up to several days before calibration and use.

- 1.4 ppm N for calibration in the lower range (concentrations less than 14 ppm N)
- 140 ppm N for calibration in the upper range (concentrations of 14 ppm N and up)

The sensor kit includes an empty bottle for this purpose.
SENSOR INSTALLATION

Unpack the ammonium sensor, hydrate it as above, and install in port 1, 2, or 3 in the sensor block at the front end of the MP TROLL 9500 as follows.

**TIP:** Remember that a polarographic D.O. sensor (if present) will operate properly only in port 2 and a turbidity wiper accessory only in port 3.

1. Remove the restrictor from the front end of the MP TROLL 9500. This allows access to the sensor block depicted in the drawing below.

2. Remove the sensor’s protective cap or storage bottle and set aside for future storage of the sensor. If the connector end is covered with a cap, remove it also.

3. Remove any moisture or dirt from the area around the port where you will install the sensor, then use the sensor removal tool to remove the plug from the port where you will install the sensor.

4. Remove any moisture or dirt from the port connector with a clean swab or tissue.

5. Check lubrication of the sensor o-rings.

**TIP:** The sensor o-rings require generous lubrication before installation. New sensors will be lubricated at the factory. If the o-rings appear dry, apply a silicone lubricant before installation.

6. Handling the sensor by the sides, not the tip, align the mark on the side of the sensor with the mark on the port.

**Avoid touching the membrane at the tip of the sensor. Contaminants on the membrane can change its properties and affect measurements.**

7. Use the sensor insertion tool to firmly press the sensor into the port until you feel it dock with the connector at the bottom. When properly inserted a small gap (width of the sensor removal tool) remains between the widest part of the sensor and the instrument body, for ease of removal.

CALIBRATION OVERVIEW

The software offers several options for ammonium calibration.

- **Three-point bithermal** ("two-temperature") calibration. The first two calibration points are taken in solutions of different concentrations at the same temperature. The third point uses one of these solutions at a temperature that is at least 10° higher or lower depending on anticipated field conditions. This type of calibration allows determination of the sensor’s “isopotential point”—the ion concentration at which changes in temperature do not cause a change in sensor response (voltage). A three-point bithermal calibration is recommended before the first use of the sensor, and regularly thereafter, to insure accurate readings at all potential temperatures.

- **Two-point isothermal** ("same temperature") calibration using solutions of two different concentrations. A two-point isothermal calibration calculates the sensor’s slope and offset but cannot compute the isopotential point. For best results this type of calibration should be carried out as close as possible to the temperature at which the sensor will be used. Or, It may be performed after a previous three-point bithermal calibration to recalculate the slope and offset of an aging sensor while retaining the previously calculated isopotential point.

- **Single-point** calibration. After the initial three-point bithermal calibration has established the sensor slope, offset, and isopotential point, a single-point calibration may be used with good results to adjust the offset on a daily basis.

CALIBRATION SOLUTIONS

Ammonium chloride (NH₄Cl) solutions certified to N.I.S.T. standards are supplied in the In-Situ Ammonium Calibration Kits:

- 14.0 ppm as N
- 140 ppm as N
- 1400 ppm as N
Specialized calibration kits are available for calibrating the ammonium sensor for low-range and high-range measurements:

- **Low range:** two quarts each 14 ppm and 140 ppm
- **High range:** two quarts each 140 ppm and 1400 ppm

### RECOMMENDED CALIBRATION FREQUENCY

Ion-selective electrodes are inherently unstable and drift is quite normal. To achieve the most accurate sensor response, we recommend a complete three-point bithermal calibration once a week, with a single-point calibration daily or after 4-6 hours of use.

### PREPARING TO CALIBRATE

You will need:

- **MP TROLL 9500** with the hydrated ammonium sensor installed and sensors or plugs in the other sensor ports
- **Cal Cup**
- One, two, or three ammonium calibration solutions, selected for the range you expect to measure. When performing a multi-point calibration, begin with the lowest-concentration solution.
- For a three-point bithermal calibration: a temperature bath, or container of ice large enough to hold the Cal Cup (and stirrer, if used).
- **Stirrer:** Use a stirrer during calibration if it will also be used in the field—for example, in stagnant or very slowly moving water. The more closely calibration conditions reflect field conditions, the more successful the calibration. For more information on the stirrer, see “Stirring” in Section 10.

### AMMONIUM CALIBRATION PROCEDURE

1. Rinse the Cal Cup and front end of the MP TROLL 9500 in clean water. Shake to dry.
   
   For best results, follow this with a rinse in a portion of the selected calibration solution. Discard the rinse solution.

2. Insure the black PVC base (or the stirrer) is attached to the Cal Cup, and fill the Cal Cup to the fill line with the selected calibration solution.
   
   - Begin with the lowest concentration when performing a multi-point calibration.
   - With a full complement of sensors installed, use the lower line as a guide.
   - With 1 or 2 removable sensors installed, fill to the upper line.

3. Insert the front end of the MP TROLL 9500 into the open end of the Cal Cup. Thread the Cal Cup onto the body until seated against the o-ring, then back off slightly to avoid overtightening.

   To stabilize the instrument, you may wish to use a calibration stand or other support.

4. Connect the MP TROLL 9500 to a PC and establish a connection in Win-Situ 4 or Pocket-Situ 4. Win-Situ screens are illustrated here. The Pocket-Situ interface is similar, with the Navigation tree at the top of the screen and the Information pane below it.

5. Select the MP TROLL 9500 in the Navigation tree.

   The software will automatically detect and display the installed sensors. If one or more sensors is installed in the wrong port, an error message will be displayed. Simply remove the sensor and install it in the correct position, then “refresh” the device before continuing.

6. Select Ammonium in the Parameters list. The sensor serial number (SN) and recent calibration information is displayed.

7. Select Calibrate.
The Ammonium Calibration Wizard starts. A screen like this is displayed.

8. Select the number of calibration points for this calibration, and the concentration (ppm) of the calibration solution for each point.

Cal point 1 is the solution the sensor is soaking in now, the lowest concentration for a multi-point calibration. If doing a three-point bithermal cal, choose cal point 3 to be the same as either cal point 1 or cal point 2.

9. Select Next to continue.

10. In the next screen, select Run to begin the stabilization.

The display will continuously update as readings are taken and compared against the stabilization criteria.

- Status indicators:
  - NOT TESTED is displayed until you begin the calibration by selecting Run.
  - UNSTABLE indicates the sensor response does not meet the criteria for a valid calibration point.
  - NOMINAL indicates the sensor deviation meets early stabilization criteria.

The Accept button becomes available when nominal stability is achieved. You may accept the early value, or wait for complete stability. If you accept the early value, the calibration point will be designated “USER SET” in the calibration report. (For more on calibration reports, see “Calibration History” in Section 10.)

STABLE is displayed when the readings have stabilized sufficiently to take a valid calibration point. The calibration proceeds automatically to the next screen.

- Sensor Reading: The current sensor response in milliVolts.
- Deviation: Change in sensor response between the last two readings.
- Current temperature is also displayed.

11. If doing a single-point calibration, go to step 16.

For a multi-point calibration, the Wizard returns to the screen shown at step 10 and waits for you to situate the probe in the next calibration solution and click Run.

12. Remove the Cal Cup, discard the first solution, rinse the Cal Cup and the front end of the instrument with clean water, followed by a rinse in the next calibration solution, refill the Cal Cup with the second solution, and attach it to the instrument.

- TIP: The used calibration solution may be flushed down the drain with running water, or saved in a separate container and used as a rinse the next time you calibrate with the same solution.

13. Select Run to begin the stabilization for cal point 2. Status indicators and controls are the same as for cal point 1 (step 10).

Again wait for Stable status (or click Accept when Nominal is indicated).

If doing a two-point calibration, go to step 16.

14. For the third calibration point, use the cal point 1 or cal point 2 solution (as specified in step 8) but change the temperature by at least 10°C. A convenient way to do this is to move the probe—Cal Cup and all—into a temperature-controlled bath or container of ice. Allow time for the sensor to reach thermal equilibration with the solution temperature. With stirring or agitation, this should take about 10 minutes, perhaps up to 30 minutes if left undisturbed.

15. When the temperature is stable, select Run for cal point 3.

When Nominal is accepted or Stable is indicated for cal point 3, the final screen is displayed.
16. The final screen of the Calibration Wizard shows the sensor slope and offset calculated during the calibration process. For a three-point bithermal calibration, the calculated isopotential point is shown. If a single-point calibration has been performed, the isopotential point is the one calculated during the last three-point bithermal calibration.

17. Select Finish to program the sensor with the newly calculated calibration coefficients.

The ammonium sensor is now calibrated and ready to use.

TIP: You can look at the calibration report right after calibrating, or at any time. See “Calibration History” in Section 10 for details.

**Options for storing sensors:**

The ammonium sensor should be calibrated immediately before use. If storage is necessary, remove the sensor from the instrument and immerse in 14 ppm N solution, for later use in the low ammonium range, or 140 ppm N solution, for use in the high range.

**SENSOR SLOPE AND OFFSET**

The expected slope for a new sensor is about 56 (± 2) mV per decade of concentration (ppm). The calibration curve begins to deviate from linear at about 1 ppm. The sensor’s zero offset is recalculated with each single-point calibration.

**UNITS AND CALCULATED MEASUREMENTS**

Ammonium ion concentration is reported in ppm (equivalent to mg/L). No calculated measurements are currently available.

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**USAGE RECOMMENDATIONS AND CAUTIONS**

**Ammonium Sensor**

**Operating Temperature**

-5°C to 40°C (23°F to 104°F) continuous temperature; can tolerate up to 50°C (122°F) intermittently

**Pressure Rating**

20 psi (14 m, 46 ft)

**pH range**

up to 8.5

**CAUTION:** Do not submerge the ammonium sensor deeper than 46 ft (14 m). Do not use in the basic pH range (8.5 or higher).

**pH**

The sensor’s pH range is that range over which a change in pH will not cause a significant change in the measured voltage. It is the plateau on a graph of pH against mV at constant concentration of the detected ion. Outside this range, a change in pH may cause a significant change in the measured mV.

**TEMPERATURE**

The higher the temperature, the shorter the lifetime of the electrode. 1°C difference in temperature causes a 2% error at 10 ppm, unless a bithermal calibration is performed.

**CONDUCTIVITY**

In saline waters (conductivities of 1,000 µS/cm or higher), the presence of interfering ions such as sodium or potassium may limit the usability of the ammonium sensor.

**POTENTIAL INTERFERENCES**

The following table lists concentrations of possible interfering ions that cause 10% error at various levels of NH₄⁺.

<table>
<thead>
<tr>
<th>Ion</th>
<th>100 ppm NH₄⁺</th>
<th>10 ppm NH₄⁺</th>
<th>1 ppm NH₄⁺</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cs⁺</td>
<td>100</td>
<td>10</td>
<td>1</td>
</tr>
<tr>
<td>K⁺</td>
<td>270</td>
<td>27</td>
<td>2.7</td>
</tr>
<tr>
<td>Ti⁺</td>
<td>3100</td>
<td>310</td>
<td>31</td>
</tr>
<tr>
<td>H⁺</td>
<td>pH 1.6</td>
<td>pH 2.6</td>
<td>pH 3.6</td>
</tr>
<tr>
<td>Ag⁺</td>
<td>270,000</td>
<td>27,000</td>
<td>2700</td>
</tr>
<tr>
<td>Li⁺</td>
<td>35,000</td>
<td>3500</td>
<td>350</td>
</tr>
<tr>
<td>Na⁺</td>
<td>11,100</td>
<td>1,100</td>
<td>110</td>
</tr>
</tbody>
</table>
SENSOR CARE AND HANDLING

SENSOR REMOVAL
Position the yoke of the sensor removal tool at the point where the sensor meets the sensor block and pry the sensor upward.

Avoid touching the membrane at the tip of the sensor. Contaminants on the membrane can change its properties and affect measurements.

MAINTENANCE/INSPECTION/CLEANING
As long as extreme pH and high organic solvent content is avoided, the sensor should last for several months at room temperature. Eventually some of the components will leach out, and this will affect the response (detection limit and scope), but this can be compensated through calibration.

If film buildup is visible on the membrane, rinse under a gentle stream of clean water, or swish gently in a mild detergent solution, rinse well with clean water, and shake to dry.

To avoid depletion of the reference solution, do not allow the sensor to soak in pure water for more than a few minutes at a time.

The electrode is not customer-refillable.

STORAGE
Store the sensor immersed in 14 or 140 ppm N solution, depending on usage requirements, rather than dry or in DI water.

REFERENCES


WHAT IS CHLORIDE?

Chloride ($\text{Cl}^-$) is a highly soluble ion in water and can potentially be present in high concentrations. Chloride enters the water supply when runoff from rain or irrigation dissolves a variety of chloride-containing salts in rock and soil. Freshwater sources, streams, lakes, and underground aquifers typically have less than 10 ppm Cl$^-$. Some waters with as little as 250 ppm Cl$^-$ will taste salty to sensitive individuals, especially if the sodium cation (Na$^+$) is also present. Wastewater from sewage or industrial facilities will normally contain higher amounts of chloride. Chloride is naturally present in higher concentrations near coastal areas where it can infiltrate canals and sewers.

WHY MEASURE CHLORIDE?

Chloride ions are not toxic to humans. However, a high chloride content may harm some structures, especially those made of metal. Chloride can increase the rate of corrosion on metals in the presence of water. Vegetation is also sensitive to the amount of chloride in the soil. Agriculturally productive soils can be turned into unproductive wastelands over a period of time by irrigating with water containing high amounts of chloride. The WHO (World Health Organization) has established 100 ppm Cl$^-$ as a maximum for water used for irrigation, while 250 ppm Cl$^-$ is the maximum for drinking water.

THE CHLORIDE SENSOR

The In-Situ sensor is an ion-selective electrode (ISE) that is selective for the chloride ion (Cl$^-$). It is a double-junction combination ISE with a silver/silver-chloride reference half-cell, solid-state sensing electrode, and reference electrolyte gel. It measures the concentration in parts per million of chloride ion in solution (ppm Cl$^-$).

SENSOR PREPARATION

To insure the chloride sensor is thoroughly hydrated, soak it in distilled water for about 15 minutes before installation. The sensor kit includes an empty bottle for this purpose. Unlike the ammonium and nitrate sensors, the solid-state chloride sensor does not require soaking in a solution of specific concentration.

SENSOR INSTALLATION

Unpack the chloride sensor, hydrate it as described above, and install in port 1, 2, or 3 in the sensor block at the front end of the MP TROLL 9500 as follows.

**TIP:** Remember that a polarographic D.O. sensor (if present) will operate properly only in port 2, and a turbidity wiper accessory only in port 3.

1. Remove the restrictor from the front end of the MP TROLL 9500. This allows access to the sensor block depicted in the drawing below.

<table>
<thead>
<tr>
<th>Typical Chloride values</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fresh water</td>
</tr>
<tr>
<td>Sea water</td>
</tr>
<tr>
<td>Irrigation water</td>
</tr>
<tr>
<td>Drinking water standard (WHO)</td>
</tr>
</tbody>
</table>
2. Remove the sensor’s protective cap or storage bottle and set aside for future storage of the sensor.

3. Remove any moisture or dirt from the area around the port where you will install the sensor, then use the sensor removal tool to remove the plug from the port where you will install the sensor.

4. Remove any moisture or dirt from the port connector with a clean swab or tissue.

5. Check lubrication of the sensor o-rings.

   TIP: The sensor o-rings require generous lubrication before installation. New sensors will be lubricated at the factory. If the o-rings appear dry, apply a silicone lubricant before installation.

6. Handling the sensor by the sides, not the tip, align the mark on the side of the sensor with the mark on the port.

7. Use the sensor insertion tool to firmly press the sensor into the port until you feel it dock with the connector at the bottom. When properly inserted a small gap (width of the sensor removal tool) remains between the widest part of the sensor and the instrument body, for ease of removal.

**CALIBRATION OVERVIEW**

The software offers several options for chloride calibration.

- **Three-point bithermal** ("two-temperature") calibration. The first two calibration points are taken in solutions of different concentrations at the same temperature. The third point uses one of these solutions at a temperature that is at least 10° higher or lower depending on anticipated field conditions. This type of calibration allows determination of the sensor’s "isopotential point" — the ion concentration at which changes in temperature do not cause a change in sensor response (voltage). A three-point bithermal calibration is recommended before the first use of the sensor, and regularly thereafter, to insure accurate readings at all potential temperatures.

- **Two-point isothermal** ("same temperature") calibration using solutions of two different concentrations. A two-point isothermal calibration calculates the sensor’s slope and offset but cannot compute the isopotential point. For best results this type of calibration should be carried out as close as possible to the temperature at which the sensor will be used. Or, it may be performed after a previous three-point bithermal calibration to recalculate the slope and offset of an aging sensor while retaining the previously calculated isopotential point.

- **Single-point** calibration. After the initial three-point bithermal calibration has established the sensor slope, offset, and isopotential point, a single-point calibration may be used with good results to adjust the offset on a daily basis.

**CALIBRATION SOLUTIONS**

Sodium chloride (NaCl) solutions certified to N.I.S.T. standards are supplied in the In-Situ Chloride Calibration Kits:

- 35.5 ppm Cl⁻
- 355 ppm Cl⁻
- 3545 ppm Cl⁻

Specialized calibration kits are available for calibrating the chloride sensor for low-range and high-range measurements:

- Low range: two quarts each 35.5 ppm and 355 ppm
- High range: two quarts each 355 ppm and 3545 ppm

**RECOMMENDED CALIBRATION FREQUENCY**

Ion-selective electrodes are inherently unstable and drift is quite normal. To achieve the most accurate sensor response, we recommend a complete three-point bithermal calibration once a week, with a single-point calibration daily or after 4-6 hours of use.

**PREPARING TO CALIBRATE**

You will need:

- MP TROLL 9500 with the hydrated chloride sensor installed and sensors or plugs in the other sensor ports
- Cal Cup
- One, two, or three chloride calibration solutions, selected for the range you expect to measure. When performing a multi-point calibration, begin with the lowest-concentration solution.
- For a three-point bithermal calibration: a temperature bath, or a container of ice large enough to hold the Cal Cup (and stirrer, if used).
- Stirrer: Use a stirrer during calibration if it will also be used in the field—for example, in stagnant or very slowly moving water. The more closely calibration conditions reflect field conditions, the more successful the calibration. For more information on the stirrer, see "Stirring" in Section 10.
CHLORIDE CALIBRATION PROCEDURE

1. Rinse the Cal Cup and front end of the MP TROLL 9500 in clean water. Rinse very thoroughly if the chloride sensor has recently been exposed to pH buffers during a pH calibration. Shake to dry.

   For best results, follow this with a rinse in a portion of the selected calibration solution. Discard the rinse solution.

2. Insure the black PVC base (or the stirrer) is attached to the Cal Cup, and fill the Cal Cup to the fill line with the selected calibration solution.

   • Begin with the lowest concentration when performing a multi-point calibration.
   • With a full complement of sensors installed, use the lower line as a guide.
   • With 1 or 2 removable sensors installed, fill to the upper line.

3. Insert the front end of the MP TROLL 9500 into the open end of the Cal Cup. Thread the Cal Cup onto the body until seated against the o-ring, then back off slightly to avoid overtightening.

   To stabilize the instrument, you may wish to use a calibration stand or other support.

4. Connect the MP TROLL 9500 to a PC and establish a connection in Win-Situ 4 or Pocket-Situ 4. Win-Situ screens are illustrated here. The Pocket-Situ interface is similar, with the Navigation tree at the top of the screen and the Information pane below it.

5. Select the MP TROLL 9500 in the Navigation tree.

   The software will automatically detect and display the installed sensors. If one or more sensors is installed in the wrong port, an error message will be displayed. Simply remove the sensor and install it in the correct position, then “refresh” the device before continuing.

6. Select Chloride in the Parameters list. The sensor serial number (S/N) and recent calibration information is displayed.

7. Select Calibrate.

   The Chloride Calibration Wizard starts. A screen like this is displayed.

8. Select the number of calibration points for this calibration, and the concentration (ppm) of the calibration solution for each point.

   Cal point 1 is the solution the sensor is soaking in now, the lowest concentration for a multi-point calibration. If doing a three-point bithermal cal, choose cal point 3 to be the same as either cal point 1 or cal point 2.

9. Select Next to continue.
10. In the next screen, select Run to begin the stabilization.

The display will continuously update as readings are taken and compared against the stabilization criteria.

- Status indicators:
  - NOT TESTED is displayed until you begin the calibration by selecting Run.
  - UNSTABLE indicates the sensor response does not meet the criteria for a valid calibration point.
  - NOMINAL indicates the sensor deviation meets early stabilization criteria.

The Accept button becomes available when nominal stability is achieved. You may accept the early value, or wait for complete stability. If you accept the early value, the calibration point will be designated “USER SET” in the calibration report. (For more on calibration reports, see “Calibration History” in Section 10.)

- STABLE is displayed when the readings have stabilized sufficiently to take a valid calibration point. The calibration proceeds automatically to the next screen.

- Sensor Reading: The current sensor response in milliVolts.
- Deviation: Change in sensor response between the last two readings.
- Current temperature is also displayed.

11. If doing a a single-point calibration, go to step 16.

   For a multi-point calibration, the Wizard returns to the screen shown at step 10 and waits for you to situate the probe in the next calibration solution and click Run.

12. Remove the Cal Cup, discard the first solution, rinse the Cal Cup and the front end of the instrument in clean water, followed by a rinse in the next calibration solution, refill the Cal Cup with the second solution, and attach it to the instrument.

   TIP: The used calibration solution may be flushed down the drain with running water, or saved in a separate container and used as a rinse the next time you calibrate with the same solution.

13. Select Run to begin the stabilization for cal point 2. Status indicators and controls are the same as for cal point 1 (step 10).

   Again wait for Stable status (or click Accept when Nominal is indicated).

   If doing a two-point calibration, go to step 16.

14. For the third calibration point, use the cal point 1 or cal point 2 solution (as specified in step 8) but change the temperature by at least 10°C. A convenient way to do this is to move the probe—Cal Cup and all—into a temperature-controlled bath or container of ice. Allow time for the sensor to reach thermal equilibration with the solution temperature. With stirring or agitation, this should take about 10 minutes, perhaps up to 30 minutes if left undisturbed.

15. When the temperature is stable, select Run for cal point 3.

   When Nominal is accepted or Stable is indicated for cal point 3, the final screen is displayed.

16. The final screen of the Calibration Wizard shows the calculated sensor slope and offset. For a three-point bithermal calibration, the isopotential point is shown. If a single-point calibration has been performed, the isopotential point is the one calculated during the last three-point bithermal calibration.

17. Select Finish to program the sensor with the newly calculated calibration coefficients.
The chloride sensor is now calibrated and ready to use.

**TIP:** You can look at the calibration report right after calibrating, or at any time. See “Calibration History” in Section 10 for details.

### Options for storing sensors:

The sensor should be calibrated immediately before use. If storage is necessary, remove the sensor from the instrument and immerse in 35 ppm Cl solution, for later use in the low chloride range, or 355 ppm Cl solution, for use in the high range.

### SENSOR SLOPE AND OFFSET

The expected slope for a new sensor is about 57 (± 2) mV per decade of concentration (ppm). The calibration curve begins to deviate from linear at about 10 ppm. The sensor’s zero offset is recalculated with each single-point calibration.

### UNITS AND CALCULATED MEASUREMENTS

Chloride ion concentration is reported in ppm (equivalent to mg/L). No calculated measurements are available.

### USAGE RECOMMENDATIONS AND CAUTIONS

**Chloride sensor**

- Operating Temperature: -5°C to 50°C (23°F to 122°F) continuous temperature; can tolerate up to 60°C (140°F) intermittently
- Pressure Rating: 100 psi (70 m, 231 ft)
- pH range: 2 to 12

**Do not submerge the chloride sensor deeper than 231 ft (70 m).**

### pH EFFECTS

The sensor’s pH range is that range over which a change in pH will not cause a significant change in the measured voltage. It is the plateau on a graph of pH against mV at constant concentration of the detected ion. Outside this range, a change in pH may cause a significant change in the measured mV.

### POTENTIAL INTERFERENCES

The table on this page lists possible interfering ions that cause 10% error at various levels of Cl⁻:

<table>
<thead>
<tr>
<th>Ion</th>
<th>100 ppm Cl⁻</th>
<th>10 ppm Cl⁻</th>
<th>1 ppm Cl⁻</th>
</tr>
</thead>
<tbody>
<tr>
<td>OH⁻</td>
<td>8000</td>
<td>800</td>
<td>80</td>
</tr>
<tr>
<td>NH₃</td>
<td>12</td>
<td>1.2</td>
<td>0.12</td>
</tr>
<tr>
<td>S₂O₃²⁻</td>
<td>1</td>
<td>0.1</td>
<td>0.01</td>
</tr>
<tr>
<td>Br⁻</td>
<td>0.3</td>
<td>0.03</td>
<td>0.003</td>
</tr>
<tr>
<td>S²⁻</td>
<td>10⁻⁴</td>
<td>10⁻⁵</td>
<td>10⁻⁶</td>
</tr>
<tr>
<td>I⁻</td>
<td>5x10⁻⁵</td>
<td>5x10⁻⁶</td>
<td>5x10⁻⁷</td>
</tr>
<tr>
<td>CN⁻</td>
<td>2x10⁻⁵</td>
<td>2x10⁻⁶</td>
<td>2x10⁻⁷</td>
</tr>
</tbody>
</table>

Complexes with Bi³⁺, Cd²⁺, Mn²⁺, Pb²⁺, Sn²⁺, Ti³⁺ and reducing agents

### SENSOR CARE AND HANDLING

#### SENSOR REMOVAL

To remove the sensor, position the yoke of the sensor removal tool at the point where the sensor meets the sensor block and pry the sensor upward.

#### MAINTENANCE/INSPECTION/CLEANING

If a film should form on the top of the membrane, use a swab to remove it, followed by a rinse in deionized water and soaking for a few minutes in a solution of 35 ppm or 355 ppm Cl.

To avoid depletion of the reference solution, do not allow the sensor to soak in pure water for more than a few minutes at a time.

The electrode is not customer-refillable.

#### STORAGE

Store the sensor immersed in 35 or 355 ppm Cl solution, depending on usage requirements. For longer-term storage, it may be rinsed and stored dry to avoid depletion of the reference solution.
WHAT IS NITRATE?

Nitrogen is an essential nutrient for plants and animals. It exists in the environment in many different forms, constantly being replenished as part of the nitrogen cycle. Nitrate (NO₃⁻) is one form of nitrogen in the ecosystem that is very soluble in water. Nitrate enters the water system when runoff from rainfall or irrigation washes through soils that contain nitrate. The nitrate dissolves in the water and is carried to nearby streams and lakes. It also permeates downward into the soil where it may enter underground aquifers. The concentration of nitrogen in a body of water depends mostly upon the land cover and soil type. Highest concentrations are associated with shallow groundwater and agricultural use of the land. Agriculture is a large contributor to the pollution of surface water and groundwater because of the use of fertilizers that contain nitrate. Also, densely populated livestock produce large quantities of manure that can be changed into nitrate upon decay.

Nitrate remains in surface water until it is consumed as a nutrient by plants or other organisms. Surface streams have nitrate concentrations ranging from 0.1 to 20 ppm N. Levels as high as 30 ppm N are found in wastewater discharges and wastewater effluent plants while levels as low as 0.05 ppm N are found in unpolluted groundwater.

WHY MEASURE NITRATE?

High nitrate levels in drinking water are associated with health problems. Nitrate is reduced to nitrite in the digestive system, where it may then oxidize iron in the hemoglobin molecule of red blood cells to form methemoglobin. Methemoglobin lacks the capacity to bind and carry oxygen to tissues. As a result of this health risk, the EPA has established 10 ppm N as the maximum limit of nitrate in drinking water for the USA.

High levels of nitrate in lakes can lead to a process called eutrophication. Nitrites are nutrients for aquatic plants and algae, causing overproduction when present in excessive levels. This accelerated growth or bloom can eventually lead to a number of negative impacts on the aquatic environment such as a reduction in dissolved oxygen, which can lead to the death of fish and other aquatic life. Reduction of sunlight to submerged aquatic vegetation due to increased coverage on the surface causes a corresponding reduction in photosynthesis and eventual death.

THE NITRATE SENSOR

The In-Situ sensor is an ion-selective electrode (ISE) that is selective for the nitrate ion (NO₃⁻). It is a double-junction combination ISE with a silver/silver-chloride reference half-cell, PVC membrane, and reference electrolyte gel. It measures the concentration in parts per million of nitrate ion in solution (calculated as nitrogen, ppm as N).

Typical Nitrate values

<table>
<thead>
<tr>
<th></th>
<th>ppm N</th>
</tr>
</thead>
<tbody>
<tr>
<td>Unpolluted groundwater</td>
<td>0.05</td>
</tr>
<tr>
<td>Surface water</td>
<td>0.1 to 20</td>
</tr>
<tr>
<td>Waste water</td>
<td>~ 30</td>
</tr>
<tr>
<td>Drinking water standard (EPA)</td>
<td>10</td>
</tr>
</tbody>
</table>
SENSOR PREPARATION

To ensure optimum membrane response, the nitrate sensor should be thoroughly hydrated in an appropriate solution before calibration. A good way to do this is to allow the sensor to soak in the solution you plan to use for the first calibration point (lowest concentration) for at least 15 minutes and up to several days before calibration and use.

- 1.4 ppm N for calibration in the lower range (concentrations less than 14 ppm N)
- 140 ppm N for calibration in the upper range (concentrations of 14 ppm N and up)

The sensor kit includes an empty bottle for this purpose.

SENSOR INSTALLATION

Unpack the nitrate sensor, hydrate it as above, and install in port 1, 2, or 3 in the sensor block at the front end of the MP TROLL 9500 as follows.

1. Remove the restrictor from the front end of the MP TROLL 9500. This allows access to the sensor block depicted in the drawing below.

   
   **TIP:** Remember that a polarographic D.O. sensor (if present) will operate properly only in port 2 and a turbidity wiper accessory only in port 3.

2. Remove the sensor’s protective cap or storage bottle and set aside for future storage of the sensor. If the connector end is covered with a cap, remove it also.

3. Remove any moisture or dirt from the area around the port where you will install the sensor, then use the sensor removal tool to remove the plug from the port where you will install the sensor.

4. Remove any moisture or dirt from the port connector with a clean swab or tissue.

5. Check lubrication of the sensor o-rings.

   **TIP:** The sensor o-rings require generous lubrication before installation. New sensors will be lubricated at the factory. If the o-rings appear dry, apply a silicone lubricant before installation.

6. Handling the sensor by the sides, not the tip, align the mark on the side of the sensor with the mark on the port.

   **Avoid touching the membrane at the tip of the sensor. Contaminants on the membrane can change its properties and affect measurements.**

7. Use the sensor insertion tool to firmly press the sensor into the port until you feel it dock with the connector at the bottom. When properly inserted a small gap (width of the sensor removal tool) remains between the widest part of the sensor and the instrument body, for ease of removal.

CALIBRATION

OVERVIEW

The software offers several options for nitrate calibration.

- **Three-point bithermal** ("two-temperature") calibration. The first two calibration points are taken in solutions of different concentrations at the same temperature. The third point uses one of these solutions at a temperature that is at least 10° higher or lower depending on anticipated field conditions. This type of calibration allows determination of the sensor’s "isopotential point"—the ion concentration at which changes in temperature do not cause a change in sensor response (voltage). A three-point bithermal calibration is recommended before the first use of the sensor, and regularly thereafter, to insure accurate readings at all potential temperatures.

- **Two-point isothermal** ("same temperature") calibration using solutions of two different concentrations. A two-point isothermal calibration calculates the sensor’s slope and offset but cannot compute the isopotential point. For best results this type of calibration should be carried out as close as possible to the temperature at which the sensor will be used. Or, it may be performed after a previous three-point bithermal calibration to recalculate the slope and offset of an aging sensor while retaining the previously calculated isopotential point.
• Single-point calibration. After the initial three-point bithermal calibration has established the sensor slope, offset, and isopotential point, a single-point calibration may be used with good results to adjust the offset on a daily basis.

**CALIBRATION SOLUTIONS**

Potassium nitrate (KNO₃) solutions certified to N.I.S.T. standards are supplied in the In-Situ Nitrate Calibration Kits:

- 14.0 ppm as N
- 140 ppm as N
- 1400 ppm as N

Specialized calibration kits are available for calibrating the nitrate sensor for low-range and high-range measurements:

- Low range: two quarts each 14.0 ppm and 140 ppm
- High range: two quarts each 140 ppm and 1400 ppm

**RECOMMENDED CALIBRATION FREQUENCY**

Ion-selective electrodes are inherently unstable and drift is quite normal. To achieve the most accurate sensor response, we recommend a complete three-point bithermal calibration once a week, with a single-point calibration daily or after 4-6 hours of use.

**PREPARING TO CALIBRATE**

You will need:

- MP TROLL 9500 with the hydrated nitrate sensor installed and sensors or plugs in the other sensor ports
- Cal Cup
- One, two, or three nitrate calibration solutions, selected for the range you expect to measure. When performing a multi-point calibration, begin with the lowest-concentration solution
- For a three-point bithermal calibration: a temperature bath, or a container of ice large enough to hold the Cal Cup (and stirrer, if used).
- Stirrer: Use a stirrer during calibration if it will also be used in the field—for example, in stagnant or very slowly moving water. The more closely calibration conditions reflect field conditions, the more successful the calibration. For more information on the stirrer, see "Stirring" in Section 10.

**NITRATE CALIBRATION PROCEDURE**

1. Rinse the Cal Cup and front end of the MP TROLL 9500 in clean water. Shake to dry.

   For best results, follow this with a rinse in a portion of the selected calibration solution. Discard the rinse solution.

2. Insure the black PVC base (or the stirrer) is attached to the Cal Cup, and fill the Cal Cup to the fill line with the selected calibration solution.

   • Begin with the lowest concentration when performing a multi-point calibration.
   • With a full complement of sensors installed, use the lower line as a guide.
   • With 1 or 2 removable sensors installed, fill to the upper line.

3. Insert the front end of the MP TROLL 9500 into the open end of the Cal Cup. Thread the Cal Cup onto the body until seated against the o-ring, then back off slightly to avoid overtightening.

   To stabilize the instrument, you may wish to use a calibration stand or other support.

4. Connect the MP TROLL 9500 to a PC and establish a connection in Win-Situ 4 or Pocket-Situ 4. Win-Situ screens are illustrated here. The Pocket-Situ interface is similar, with the Navigation tree at the top of the screen and the Information pane below it.

5. Select the MP TROLL 9500 in the Navigation tree.

   The software will automatically detect and display the installed sensors. If one or more sensors is installed in the wrong port, an error message will be displayed. Simply remove the sensor and install it in the correct position, then "refresh" the device before continuing.
6. Select Nitrate in the Parameters list. The sensor serial number (SN) and recent calibration information is displayed.

7. Select **Calibrate**.

The Nitrate Calibration Wizard starts. A screen like this is displayed.

8. Select the number of calibration points for this calibration, and the concentration (ppm) of the calibration solution for each point.

   Cal point 1 is the solution the sensor is soaking in now, the lowest concentration for a multi-point calibration. If doing a three-point bithermal cal, choose cal point 3 to be the same as either cal point 1 or cal point 2.

9. Select **Next** to continue.

10. In the next screen, select **Run** to begin the stabilization.

The display will continuously update as readings are taken and compared against the stabilization criteria.

- **Status indicators:**
  - **NOT TESTED** is displayed until you begin the calibration by selecting Run.
  - **UNSTABLE** indicates the sensor response does not meet the criteria for a valid calibration point.
  - **NOMINAL** indicates the sensor deviation meets early stabilization criteria.

The **Accept** button becomes available when nominal stability is achieved. You may accept the early value, or wait for complete stability. If you accept the early value, the calibration point will be designated “USER SET” in the calibration report. (For more on calibration reports, see “Calibration History” in Section 10.)

- **STABLE** is displayed when the readings have stabilized sufficiently to take a valid calibration point. The calibration proceeds automatically to the next screen.
  - **Sensor Reading:** The current sensor response in milliVolts.
  - **Deviation:** Change in sensor response between the last two readings.
  - **Current temperature** is also displayed

11. If doing a single-point calibration, go to step 16.

   For a multi-point calibration, the Wizard returns to the screen shown at step 10 and waits for you to situate the probe in the next calibration solution and click Run.
12. Remove the Cal Cup, discard the first solution, rinse the Cal Cup and the front end of the instrument with clean water, followed by a rinse in the next calibration solution, refill the Cal Cup with the second solution, and attach it to the instrument.

**TIP:** The used calibration solution may be flushed down the drain with running water, or saved in a separate container and used as a rinse the next time you calibrate with the same solution.

13. Select **Run** to begin the stabilization for cal point 2. Status indicators and controls are the same as for cal point 1 (step 10).

Again wait for Stable status (or click Accept when Nominal is indicated).

If doing a two-point calibration, go to step 16.

14. For the third calibration point, use the cal point 1 or cal point 2 solution (as specified in step 8) but change the temperature by at least 10°C. A convenient way to do this is to move the probe—Cal Cup and all—into a temperature-controlled bath or container of ice. Allow time for the sensor to reach thermal equilibration with the solution temperature. With stirring or agitation, this should take about 10 minutes, perhaps up to 30 minutes if left undisturbed.

15. When the temperature is stable, select **Run** for cal point 3.

When Nominal is accepted or Stable is indicated for cal point 3, the final screen is displayed.

16. The final screen of the Calibration Wizard shows the sensor slope and offset calculated during the calibration process. For a three-point bithermal calibration, the calculated isopotential point is shown. If a single-point calibration has been performed, the isopotential point is the one calculated during the last three-point bithermal calibration.

17. Select **Finish** to program the sensor with the newly calculated calibration coefficients.

The nitrate sensor is now calibrated and ready to use.

**TIP:** You can look at the calibration report right after calibrating, or at any time. See “Calibration History” in Section 10 for details.

**Options for storing sensors:**

The sensor should be calibrated immediately before use. If storage is necessary, remove the sensor from the instrument and immerse in 14 ppm N solution, for later use in the low nitrate range, or 140 ppm N solution, for use in the high range.

**SENSOR SLOPE AND OFFSET**

The expected slope for a new sensor is about 57 (± 2) mV per decade of concentration (ppm). The calibration curve begins to deviate from linear at about 10 ppm. The sensor’s zero offset is recalculated with each single-point calibration.

**UNITS AND CALCULATED MEASUREMENTS**

Nitrate ion concentration is reported in ppm (equivalent to mg/L). No calculated measurements are available.

**USAGE RECOMMENDATIONS AND CAUTIONS**

Nitrate Sensor

Operating Temperature  
-5°C to 40°C (23°F to 104°F) continuous temperature; can tolerate up to 50°C (122°F) intermittently

Pressure Rating  
20 psi (14 m, 46 ft)

pH range  
2.5 to 11

**TEMPERATURE**

The higher the temperature, the shorter the lifetime of the electrode. 1°C difference in temperature causes a 2% error at 10 ppm, unless a bithermal calibration is performed.
POTENTIAL INTERFERENCES

The following table shows concentrations of possible interfering ions that cause 10% error at various levels of NO₃⁻.

<table>
<thead>
<tr>
<th>Ion</th>
<th>100 ppm NO₃⁻</th>
<th>10 ppm NO₃⁻</th>
<th>1 ppm NO₃⁻</th>
</tr>
</thead>
<tbody>
<tr>
<td>ClO₄⁻</td>
<td>0.01</td>
<td>0.001</td>
<td>0.0001</td>
</tr>
<tr>
<td>I⁻</td>
<td>0.5</td>
<td>0.05</td>
<td>0.005</td>
</tr>
<tr>
<td>ClO₃⁻</td>
<td>5</td>
<td>0.5</td>
<td>0.05</td>
</tr>
<tr>
<td>CN⁻</td>
<td>10</td>
<td>1</td>
<td>0.1</td>
</tr>
<tr>
<td>Br⁻</td>
<td>70</td>
<td>7</td>
<td>0.7</td>
</tr>
<tr>
<td>NO₂⁻</td>
<td>70</td>
<td>7</td>
<td>0.7</td>
</tr>
<tr>
<td>HS⁻</td>
<td>100</td>
<td>10</td>
<td>1</td>
</tr>
<tr>
<td>HCO₃⁻</td>
<td>1000</td>
<td>100</td>
<td>10</td>
</tr>
<tr>
<td>CO₃²⁻</td>
<td>2000</td>
<td>200</td>
<td>20</td>
</tr>
<tr>
<td>Cl⁻</td>
<td>3000</td>
<td>300</td>
<td>30</td>
</tr>
<tr>
<td>H₂PO₄⁻</td>
<td>5000</td>
<td>500</td>
<td>50</td>
</tr>
<tr>
<td>HPO₄²⁻</td>
<td>5000</td>
<td>500</td>
<td>50</td>
</tr>
<tr>
<td>PO₄³⁻</td>
<td>5000</td>
<td>500</td>
<td>50</td>
</tr>
<tr>
<td>AcO⁻</td>
<td>20,000</td>
<td>2000</td>
<td>200</td>
</tr>
<tr>
<td>F⁻</td>
<td>60,000</td>
<td>6000</td>
<td>600</td>
</tr>
<tr>
<td>SO₄²⁻</td>
<td>100,000</td>
<td>10,000</td>
<td>1000</td>
</tr>
</tbody>
</table>

SENIOR CARE AND HANDLING

SENSOR REMOVAL

Position the yoke of the sensor removal tool at the point where the sensor meets the sensor block and pry the sensor upward.

Avoid touching the membrane at the tip of the sensor. Contaminants on the membrane can change its properties and affect measurements.

MAINTENANCE/INSPECTION/CLEANING

As long as extreme pH and high organic solvent content is avoided, the sensor should last for several months at room temperature. Eventually some of the components will leach out, and this will affect the response (detection limit and scope), but this can be compensated through calibration.

If film buildup is visible on the membrane, rinse under a gentle stream of clean water, or swish gently in a mild detergent solution, rinse well with clean water, and shake to dry.

To avoid depletion of the reference solution, do not allow the sensor to soak in pure water for more than a few minutes at a time.

The electrode is not customer-refillable.

STORAGE

Store the sensor immersed in 14 or 140 ppm N solution, depending on usage requirements, rather than dry or in DI water.

REFERENCES


WHAT IS TURBIDITY?

Turbidity is an indirect measure of the clarity or transparency of water, and thus an important indicator of its condition and productivity. Created by suspended matter and microscopic organisms, turbidity causes light to be scattered and absorbed rather than transmitted directly through water. Turbidity is the physical characteristic of the solution that causes light scattering. Turbidity is the opposite of clarity.

The APHA reference work Standard Methods (Eaton and others, 2005) defines turbidity as “an expression of the optical property that causes light to be scattered and absorbed rather than transmitted with no change in direction or flux level through the sample.”

Turbidity is not . . .

• a direct measure of clarity.
• a measure of color.
• a measure of suspended solids; it is a measure of their light-scattering abilities.

The In-Situ Turbidity sensor* is in conformance with the ISO 7027 turbidity standard which specifies 90° scattered light.

WHY MEASURE TURBIDITY?

Turbidity measurements

• can provide a reasonable estimate of the total suspended solids or sediments (TSS) concentration in water.
• can tell us something about the health of a natural water body. Clear water lets light penetrate more deeply into a lake or stream than does murky water. This light allows photosynthesis to occur and oxygen to be produced.

Typical turbidity values

<p>| | |</p>
<table>
<thead>
<tr>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>EPA drinking water</td>
<td>0.3 - 0.5 NTU</td>
</tr>
<tr>
<td>Treated water</td>
<td>0 - 1 NTU</td>
</tr>
<tr>
<td>Fresh water, &gt;21.5” visibility</td>
<td>&lt; 10 NTU</td>
</tr>
<tr>
<td>Fresh water, 2.5” visibility</td>
<td>240 NTU</td>
</tr>
<tr>
<td>Short-term stress to aquatic life</td>
<td>&gt; 10 NTU</td>
</tr>
<tr>
<td>Unsafe level for most aquatic life</td>
<td>&gt; 100 NTU</td>
</tr>
</tbody>
</table>

• can be a useful indicator of runoff into surface water systems.
• in flow-cell or in-line applications, when pumping water at very low rates, can provide a good indication of true formation water.

Higher turbidity levels make it more costly to treat surface water for use as drinking water. Controlling turbidity may be an effective way to protect against pathogens in drinking water.

Aesthetic considerations also play a role in our desire to quantify turbidity: Most people would rather look at, drink, or swim in clear water than in water that appears cloudy, and closely associate appearance with the health of the body of water.

HOW IS TURBIDITY MEASURED?

Historical methods for measuring turbidity relied on subjective estimates that depended largely on the eye of the beholder.

In the Jackson Candle method, for example, a candle flame is observed through the length of a glass tube into which a fluid sample is poured until the rays of transmitted and scattered light appear equal and the flame essentially disappears. Among several drawbacks to this method, the reproducibility of standards formulated from natural sediments was difficult to control.

The Secchi disk method used in limnological studies involves submerging a weighted, black-and-white painted metal plate until the pattern can no longer

* Contains an assembly, US Pat. No. 6,842,243
be detected. The plate is then pulled up until it is visible again. The average of the two depths provides an estimate of water clarity or transparency.

Modern turbidimeters measure the loss in intensity of a light beam as it passes through a solution containing suspended and dissolved particles that are large enough to scatter the light. The method is based upon a comparison of the intensity of light scattered by the sample with the intensity of light scattered by a standard reference suspension. The nephelometer is a particular type of turbidimeter that measures the intensity of light scattered at right angles (90°) to the incident light. This lessens the difficulty of differentiating small changes against a large background. Standards for turbidity-measurement instruments specify the light source, angle, wavelength, beam width, and sample suspensions, among other factors. Many of today's commonly accepted procedures (e.g., Standard Methods, EPA, and ISO) apply to laboratory bench-top instruments.

THE TURBIDITY SENSOR

The optional turbidity sensor of the Multi-Parameter TROLL 9500 is permanently-installed and factory-calibrated. It may be a turbidity sensor alone or a pressure/turbidity sensor combination. If your MP TROLL 9500 was ordered without a turbidity sensor or a pressure sensor, there will be a permanently installed plug in the pressure/turbidity sensor slot. A turbidity sensor or combination pressure/turbidity sensor can be added at the factory.

The In-Situ turbidity sensor is comprised of a matched solid-state detector-emitter pair positioned at right angles. The light source is an infrared LED, optimized for operation at 870 nanometers (nm). The optical windows of the detector (photodiode) and emitter (LED) are scratch-resistant sapphire. ISO 7027 has set a detection angle of 90° and the light wavelength at 860 nm. The sensor uses active modulation for ambient light rejection.

The In-Situ sensor is an electronic nephelometer which compares the intensity of light scattered by the environmental fluid with intensity of light scattered by a standard reference suspension. The higher the intensity of scattered light, as measured in NTU's, the higher the turbidity. This measurement generally provides a very good correlation with the concentration of particles in the water that affect clarity. However, measurements of scattered light cannot be directly related to a gravimetric equivalent, such as suspended sediment load, unless a working curve for the specific sample is created.

THE TURBIDITY WIPER

The optional wiper accessory helps to keep the turbidity sensor optics free of bubbles and fouling.

The wiper installs in port 3 of the MP TROLL 9500 like other removable sensors. A positional brace aligns it with respect to the turbidity sensor and keeps it stable in moving waters.

The wiper pad is adhesive-free, low-abrasion cotton material. The pad is easily replaced when it becomes too soiled to clean the sensor optics effectively.

⚠️ Use of the wiper will significantly impact battery life. Lithium batteries are recommended.
WIPE INSTALLATION

The MP TROLL 9500 may be shipped with the optional wiper already installed in port 3, as shown on the drawing below. If installation is necessary, unpack and install the wiper in port 3 as follows.

1. Remove the restrictor from the front end of the MP TROLL 9500. This allows access to the sensor block depicted in the drawing below.

2. Use the sensor removal tool to remove the sensor or plug from port 3. Retain the plug for future use. For best access to port 3, you may need to remove sensors or plugs from other ports as well.

3. Check lubrication of the o-rings on the connector end of the wiper.

4. Visually align the connector on the wiper with the connector at the bottom of port 3.

5. Press firmly until you feel the wiper dock with the port connector. When properly inserted a small gap (width of the sensor removal tool) remains between the wiper body and the instrument body.

6. Press the wiper into the bracket attached to the turbidity sensor.

7. After installing a new wiper, we recommend you access wiper control in the software while you can clearly see the wiper movement. Connect in software, select the wiper, and click Wipe to ensure the wiper passes over the turbidity sensor optics properly.

WIPE MOVEMENT

When the turbidity sensor is off—not taking a measurement—the wiper head is "parked" over port 4. When a turbidity measurement is requested, the wiper head passes over the optics, sweeping them clean, and returns to its parking place. One "wipe" consists of a 360° counter-clockwise sweep (viewed from the sensor end), as shown in the drawing below. Wiping occurs automatically before turbidity readings—manual reads, profiling, calibration, and tests—that are more than 15 seconds apart. If readings are less than 15 seconds apart, the wiper will wipe once, before the first reading.

A single wipe may be initiated in the software when the instrument is idle to clear the turbidity optics of bubbles or debris. The wiper’s movements are entirely software-controlled.

TIP: 15 seconds are allotted for a wipe cycle. This time is generous to allow for slower wiper movement at very low temperatures.

If readings—test, calibration, profiling—are more than 15 seconds apart, the turbidity sensor will be wiped automatically before each reading. If readings are less than 15 seconds apart, the wiper will wipe the windows just once, before the first reading.
MANUAL WIPE

To wipe the turbidity sensor optics manually:

1. With the wiper installed in port 3, connect the MP TROLL 9500 to a PC and establish a connection in Win-Situ 4 or Pocket-Situ 4.
2. Select the Wiper in the Navigation tree.
3. Click Wipe. The wiper will pass once over the turbidity sensor optics and return to its home position.

WIPER GUIDELINES AND PRECAUTIONS

• If a wiper is installed during a Quick Cal of the Basic sensors (see Section 3, Getting Started), steps should be taken to insure the wiper pad material does not absorb the Quick Cal solution. There are two ways to do this.
  – Remove the wiper head before doing the Quick Cal. Refer to Wiper Maintenance guidelines later in this section for instructions on removing the wiper head.
  – Alternatively, soak the front end of the instrument in plain water before calibrating to allow the wiping pad to absorb sufficient water to prevent its absorbing any Quick Cal solution.

• Do not attempt to move the wiper head by hand. Wiper movement is software-controlled.

• The wiper pressure may be adjusted if necessary so that the pad is effectively cleaning the sapphire windows of the turbidity sensor during movement. Refer to Wiper Maintenance guidelines later in this section.

• The wiper pad or head may be replaced as needed. Refer to Wiper Maintenance guidelines later in this section.

• When an RDO optical dissolved oxygen sensor is installed, check to see that the RDO adapter cable is out of the way of wiper movement.

CALIBRATION

FACTORY CALIBRATION

The turbidity sensor has been factory-calibrated to achieve a sensor accuracy of ± 5% or 2 NTU (whichever is greater). The sensor is calibrated over its full range, 0 to 2000 NTU, using polymer standards. The resulting calibration coefficients are written to the sensor memory, where they are stored permanently. They may be overlaid by performing a field calibration as described below, or may be recalled from the sensor memory at any time.

The MP TROLL 9500’s turbidity sensor is ready to measure turbidity without any user intervention. It is advisable to take a turbidity reading first in your own calibration solution(s) as a check to ensure the accuracy is within your operational standards and requirements. If this result is satisfactory, a field calibration is not required.

FIELD CALIBRATION

Field calibration (or “user calibration”) is an overlay function that is applied after the factory calibration math is done. The factory calibration applies across the entire range of NTU, and can be altered in the field with a 1 to 4 point calibration procedure as described below to compensate for effects of sensor fouling and other factors. You may wish to perform a field calibration with standards other than polymer (i.e., Formazin).

For best results, calibrate as close to field temperature as possible.

CALIBRATION SOLUTIONS

A nephelometer such as the In-Situ turbidity sensor should be calibrated using standard reference suspensions having reproducible light-scattering properties. The sensor has been factory-calibrated with polymer suspensions, and the resulting calibration coefficients take into account the light-scattering properties of the suspensions and the sensor optics.

Why do I need to calibrate the turbidity sensor if it has been calibrated in the factory?

A new turbidity sensor is ready to measure turbidity with reference to suspended polymer standards. If you prefer to reference turbidity measurements to Formazin rather than polymer, a field calibration with Formazin should be performed.

After cleaning the sensor, readings should be checked with standards and a field calibration performed if necessary.
Polymer-based standards are submicron, non-surface charged, solid spheres in matrixes of ultrapure water in homogeneous suspension; this homogeneity allows linear dilutions. EPA approved the polymer suspensions in 1984 as a calibration standard for turbidimeters. Polymer suspensions are very stable, can withstand temperature extremes, and have excellent lot-to-lot precision. A range of polymer suspensions are available individually from In-Situ Inc. for calibrating any expected turbidity range. They can be diluted with turbidity-free water to achieve other NTU values (but see the cautions below).

Avoid vigorous mixing or agitation, which will create air bubbles and lower the accuracy of the standards. It is advantageous to calibrate a nephelometric turbidimeter with a standard that most closely matches the size of the particulates you will be measuring.

**Calibrating with Other Solutions**

**Formazin:** If you wish to recalibrate with Formazin, keep these points in mind.

- Requires very careful handling.
- Must be shaken gently and allowed to settle for at least 5 minutes before use.
- Should not be diluted.

**Diluting Polymer Suspensions:** If you dilute polymer suspensions, keep these points in mind:

- Do not dilute more than 10:1. Use good laboratory techniques.
- Store carefully. PVC bottles are recommended.
- Diluting polymer suspensions takes them out of the category of “primary standards”, they become “secondary standards.”
- If not handled carefully, the dilutions can become unstable; the suspension of particles may be lost.

**RECOMMENDED CALIBRATION FREQUENCY**

Your own experience is the best guide to how often the turbidity sensor will benefit from recalibration. The need for recalibration depends on the condition of the optical windows, which in turn depends on the environment. In a biologically active environment, cleaning and calibration will be required more often. Periodic checks in calibration solutions of known turbidity can be beneficial in indicating how well the sensor is holding its calibration.

A field calibration is recommended

- if you wish to reference turbidity measurements to a standard other than polymer.
- after cleaning the sensor windows of contamination.
- when readings appear to drift, or show unexpectedly high or low results.
- if algal or other growth on the front end of the MP TROLL 9500 changes the reflective properties of the device.

**TURBIDITY CALIBRATION PROCEDURE**

You will need:

- MP TROLL 9500 with turbidity sensor (wiper optional), plugs in any unused sensor ports.
- The restrictor, nose cone, and removable sensors (if any) that will be installed when turbidity is measured.
- A laboratory beaker large enough to hold the instrument and calibration solution.
- One or more calibration standards for the region in which you wish to calibrate. Several ranges from Very Low to Full are suggested in the Calibration Wizard.

**TIP:** A 1-point to 4-point field calibration may be carried out in any range. When performing a multi-point calibration, begin with 0 NTU solution. For best measurement precision the highest NTU value should exceed the readings you expect in the field.

1. Rinse the front end of the MP TROLL 9500 with clean water. Shake well to remove the rinse water; dry external surfaces (not the optical windows) with a clean tissue.

2. Pour the selected calibration standard into the beaker and insert the MP TROLL 9500 into the solution.

The windows of the turbidity sensor should be immersed at least ¼” (a quarter of an inch) deep in the solution. If no wiper is present, gently agitate the instrument to dispel any air bubbles.
3. Connect the MP TROLL 9500 to a PC and establish a connection in Win-Situ 4 or Pocket-Situ 4. Win-Situ screens are illustrated here. The Pocket-Situ interface is similar, with the Navigation tree at the top of the screen and the Information pane below it.

4. Select the MP TROLL 9500 in the Navigation tree.

The installed sensors will be displayed—including the turbidity wiper, if installed in port P3.

5. Click to select Turbidity in the Parameters list. The sensor serial number (S/N) and recent calibration information is displayed.

6. Select Calibrate.

The Turbidity Calibration Wizard starts. A screen like the one below is displayed.

7. Select the type of calibration you wish to do:

   - Use Default Coefficients resets the factory defaults. If you select this option, click Next and proceed to step 17.

   - Single-Point Calibration. Click Next and go to step 12.

   - Standard Calibration (default).

8. If you selected a Standard Calibration, select an operational range target. The ranges are suggestions only; a 1- to 4-point calibration may be performed in any range, using any standards

   Note: If the software detects a turbidity wiper accessory, pressing the Wipe button will result in one complete wipe cycle of the turbidity sensor optics.

9. Select Next to continue.

10. In the next screen, select the number of calibration points for this calibration, and the turbidity value (in NTU) of the calibration solution for each point. One to four points (solutions) may be selected for any operational range target selected in the previous screen.

11. Select Next to continue.

   When performing a multi-point calibration, cal point 1 must be taken in a standard with a value of 0 NTU. Use clear water for this. Purchased distilled or deionized water will generally measure less than 0.5 NTU. Filtered water will have a lower NTU value.
A screen similar to the one shown below is displayed.

![Screen Display](image)

12. If you are performing a single-point calibration, enter the value of the calibration standard (NTU). For a multi-point calibration, the value of the first solution will be displayed.

13. When the sensor is situated in the calibration medium, select **Run** to begin the stabilization.

   The display will continuously update as readings are taken and compared against the stabilization criteria.

   • **Status indicators:**
     - **NOT TESTED** is displayed until you begin the calibration by selecting Run.
     - **UNSTABLE** indicates the sensor response does not meet the criteria for a valid calibration point.
     - **NOMINAL** indicates the sensor deviation meets early stabilization criteria.

     The **Accept** button becomes available when nominal stability is achieved. You may accept the early value, or wait for complete stability. If you accept the early value, the calibration point will be designated “USER SET” in the calibration report. (For more on calibration reports, see “Calibration History” in Section 10.)

     **STABLE** is displayed when the readings have stabilized sufficiently to take a valid calibration point. The calibration proceeds automatically to the next screen.

   • **Sensor Reading:** The current sensor response in NTU
   • **Sensor Deviation:** Change in sensor response between the last two readings.
   • **Temperature**

14. If doing a one-point calibration, go to step 17.

   For a multi-point calibration, the Wizard returns to the screen shown at step 13 and waits for you to situate the probe in the next calibration solution and click Run.

15. Discard the first solution, rinse the beaker and the front end of the instrument thoroughly, wipe off excess water, refill the beaker with the second solution, and insert the MP TROLL 9500 as before.

16. Select **Run** to begin the stabilization for the second calibration point. Status indicators and controls are the same as for the first calibration point (step 13).

   Again wait for stabilization, dump, rinse, dry, refill, Run, as many times as necessary to collect a stable calibration point in each solution.

17. The final screen shows the sensor slope and offset calculated during the calibration process (or the default settings if you selected that option at step 7). A slope and offset will be shown for each calibration point.

   “Pivot” designates the point at which the slope characteristics change with a multi-point calibration. The correct slope for the turbidity values being monitored will automatically be applied.

18. Select **Finish** to program the sensor with the newly calculated calibration coefficients.

   **TIP:** You can look at the calibration report right after calibrating, or at any time. See “Calibration History” in Section 10 for details.
RESETTING DEFAULT COEFFICIENTS

The sensor’s calibration may be reset back to factory defaults at any time. As long as there is no contamination on the optical windows, this will restore the factory accuracy (± 5% or 2 NTU).

1. Establish a connection to the instrument in Win-Situ 4 or Pocket-Situ 4.
2. Select Turbidity in the Parameters list and click Calibrate.
3. In the first screen, select Use Default Coefficients, then Next.
4. In the final screen, click Finish to restore the sensor’s factory calibration coefficients.

SENSOR SLOPE AND OFFSET

The offset is factory-set at 0 NTU. The zero offset may be recalculated for any appropriate value by performing a single-point calibration using a calibration standard of the desired NTU value. The sensor response is very linear up to 200 NTU.

UNITS AND CALCULATED MEASUREMENTS

Two units are available for readings from the turbidity channel:
- NTUs—Nephelometric Turbidity Units. Select NTU when the sensor has been calibrated with polymer suspensions.
- FNUs—Formazin Turbidity Units. Select FNU when the sensor has been calibrated with Formazin.

USAGE RECOMMENDATIONS AND CAUTIONS

The operational pressure rating of the turbidity sensor is 150 psi. Do not submerge it deeper than 346 ft (105 m).

Avoid use of the stirrer accessory (recommended for monitoring dissolved oxygen in stagnant water) when measuring turbidity.

When used without a wiper, dirty sensor optics can be compensated for to some extent by changing the offset.

Optical absorbancy (“color”) will lessen the turbidity signal.

Turbidity readings are temperature-compensated.

The optics need 5 seconds warm-up time to take the first reading later. Subsequent readings can be returned instantaneously.

COMMON INTERFERENCES

Light scattering depends upon the size, shape, refractive index, and other characteristics of the particles and the wavelength of the light.

Optically black particles, such as those of activated carbon, may absorb light and effectively decrease turbidity measurements.

Nephelometers are relatively unaffected by small changes in design parameters and therefore are specified as the standard instrument for measurement of low turbidities. Nonstandard turbidimeters, such as forward-scattering devices, are more sensitive than nephelometers to the presence of larger particles and are useful for process monitoring. Reported turbidities are heavily dependent on the particulate matter contained in the suspensions that are used to prepare instrument calibration curves.

Due to current technological limitations, field turbidity measurement is “a snapshot of averages,” Field measurements can be an excellent indicator of in-situ turbidity; final determination for reporting purposes should be conducted in a laboratory.

PROFILING TURBIDITY

The turbidity sensor’s 5-second warmup will result in a slight delay before the first Profiler reading for all parameters. Subsequent readings can be taken within the Profiler’s 2-second cycling.

If a turbidity wiper accessory is installed, it performs an initial wipe of the sensor optics—this takes about 15 seconds—then displays the first turbidity reading. If the profiling rate is longer than 15 seconds, this 15 second wipe will happen before each reading. To avoid this delay, set the profiling rate to less than 15 seconds. See Customizing the Profiler in Section 5 for details.

LOGGING TURBIDITY DATA

The wiper is activated automatically before turbidity readings during tests, so long as the readings are 15 seconds or more apart. To prolong battery life when running a wiper, we recommend the use of external power or two internal lithium D-cells installed in the MP TROLL 9500.

SENSOR CARE

INSPECTION/MAINTENANCE/CLEANING

The optical windows of the sensor are made of scratch-resistant sapphire. The optical components are not user-serviceable. Serious mechanical and temperature shock are about the only things that can damage the LED. If you feel the instrument has suffered such damage, contact In-Situ Technical Support.

However, the windows may need frequent cleaning, especially if used in a biologically active environment. A wiper accessory can help to prevent the accumulation of foreign material.
Cleaning may be necessary if the optical windows of the sensor become visibly contaminated by the gradual accumulation of foreign material. Because the sensor is not removable, we recommend gentle swabbing of the windows with a circular motion using plain water. Solvents are not recommended, although an ammonia solution (e.g., grocery-store ammonia) may be used with good effect to remove particularly stubborn materials.

A calibration check should be performed after cleaning, using calibration standards.

**WIPER MAINTENANCE**

The cotton wiper pad will require replacement periodically to maintain its effectiveness in cleaning the turbidity sensor optics. The entire head may be replaced, or just the pad. In either case, the wiper head will need to be removed. A hex wrench is supplied for this purpose. You do not need to remove the entire wiper; leave the wiper body installed in port 3.

Replacement pads and wiper heads are available from In-Situ Inc. or your distributor.

**Removing the Wiper Head**

With the wiper parked over port 4, loosen the set screw on the wiper head until you can grasp the wiper head and gently pull it out.

**Replacing the Wiper Pad**

1. Remove the wiper head as above. Remove and discard the used pad.

2. Insert a new pad into the slots with the smooth side facing out, and pull to eliminate slack. Excess material may be trimmed close to the wiper head.

3. Position the head on the motor shaft with the pad facing down toward the sensors. The button at the top of the shaft should be flush with the wiper head surface. Tighten the set screw against the flat of the motor shaft. **Be very careful not to move the wiper head in a lateral direction by hand after tightening.**

4. If convenient, connect in software, select the wiper, and click **Wipe** to ensure the wiper passes over the turbidity sensor optics properly.

**Adjusting Wiper Pressure**

If necessary, loosen the set screw on the wiper head and gently pull the head up or press down lightly to ensure the pad just brushes the optical windows when it passes over the turbidity sensor. Then retighten the set screw. **Be very careful not to move the wiper head in a lateral direction by hand when engaged with the motor shaft.**

**TIP:** The wiper head is at the best height when it just brushes the optical windows of the turbidity sensor—too high and it will not clean effectively; too low and it may not be able to spin.

If it is necessary to remove the entire wiper assembly, be sure to use the sensor removal tool and grasp the body of the wiper. Do not attempt to pull the wiper out by the head.

**Wiper Replacement Parts**

<table>
<thead>
<tr>
<th>Catalog No.</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>0044520</td>
<td>Replacement wiper head</td>
</tr>
<tr>
<td>0044530</td>
<td>Replacement wiper pad replacement kit</td>
</tr>
</tbody>
</table>

**REFERENCES**

ASTM method D1889-88(A)


SDI-12 is a serial digital interface that operates at 1200 baud. In-Situ’s SDI-12 Adapter enables processing of the MP TROLL 9500’s sensor measurements by a standard SDI-12 data recorder.

**SDI-12 REQUIREMENTS**
- MP TROLL 9500 (firmware 1.54 or later)
- SDI-12 Adapter (for proper power management)
- SDI-12 data recorder (customer-supplied)

**WIRING**
Connect the stripped and tinned wires from the SDI-12 Adapter to the terminal block of an SDI-12 data recorder (or to an SDI-12 network) as follows.
- WHITE - serial data line
- BLACK - ground line
- RED - 12-volt supply line
- GREEN - shield

The data recorder or an external power supply may provide power (9.6 - 16 V) to the 12V line. The shield should be terminated (grounded) at the data recorder.

Minimum voltage supplied per the SDI-12 specification (9.6V) is sufficient to power a TROLL 9500 on 170 ft (52 m) of RuggedCable. Cables as long as 300 ft (91 m) may be used, depending on power supplied. Before using cables longer than 170 ft (52 m) we recommend that you measure the voltage at the Adapter. The table lists power requirements for specific submersible cable lengths.

**CONNECTIONS**
Attach the Twist-Lock Connector on the MP TROLL’s RuggedCable to the matching connector on the SDI-12 Adapter.

The Adapter is weather-resistant but not completely waterproof. It is not designed to be exposed to the elements. Provide a weather-resistant enclosure for optimum operation.

### Table 19-1. Cable length & power supply requirements

<table>
<thead>
<tr>
<th>Cable length</th>
<th>Power supply requirement</th>
</tr>
</thead>
<tbody>
<tr>
<td>Feet</td>
<td>meters</td>
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<tr>
<td>170</td>
<td>52</td>
</tr>
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<td>180</td>
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<td>190</td>
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<td>290</td>
<td>88</td>
</tr>
<tr>
<td>300</td>
<td>91</td>
</tr>
</tbody>
</table>

**SDI-12 SUPPORT**
The MP TROLL 9500 supports the SDI-12 Version 1.3 commands. Data loggers that support SDI-12 Version 1.3 can usually send the Version 1.3 commands to an SDI-12 “sensor” like the MP TROLL automatically. These commands are listed later in this section. Additional information may be found in an SDI-12 reference, such as that listed at the end of this section. Or consult your SDI-12 data logger documentation for more specific information.

- Insure SDI-12 is enabled (factory default) on the 9500. In Win-Situ 4 or Pocket-Situ 4, select the TROLL, click Edit... select SDI-12 Mode Preferences.
- Calibration of the water-quality sensors will need to be done through Win-Situ 4 or Pocket-Situ 4 as SDI-12 protocols do not support calibration.
**SENSOR IDENTIFICATION**

In response to the “send identification” command, the MP TROLL 9500 will respond as follows:

The default sensor address is 0. The device supports software-changeable addresses.

```
013IN-SITU WQP100154030452 <CR><LF>
```

**SENSOR VERIFICATION**

In response to the Verification command, the MP TROLL 9500 will respond as follows:

```
0+030452+30+1.54+3.0+5+3.0+4.5 <CR><LF>
```

**MAKING SDI-12 MEASUREMENTS**

When measurements are made they will be taken on all sensors installed in the MP TROLL 9500. “Derived” or calculated measurements, available in Win-Situ, are reported in SDI-12 for D.O. and conductivity. For example, the D.O. sensors can report oxygen concentration in milligrams per liter and saturation in percent. Specific conductance is reported as well as conductivity.

**TIP:** SDI-12 protocols use the term “sensor” for a single SDI-12 device with an address. In this usage, the MP TROLL 9500 is a “sensor” even though it includes up to 11 installed “sensors” in the In-Situ usage. The “sensors” in the In-Situ usage cannot be addressed individually using SDI-12 protocols.

The default order in which parameters are reported, and the units for each parameter, are listed in Table 19-2.

According to the SDI-12 specification the Start Measurement command “aM!” can take up to 9 measurements. Since the MP TROLL 9500 can potentially report more than 9 measurements there are two approaches to measuring all sensors:

- Use the Start Concurrent Measurement command (“aC!”). This command supports up to 20 measurements.

- Use the Start Additional Measurement commands (“aMn!”). The logger would first issue an “aM1!” command. If the command responds with 9 measurements then the logger will send an “aM2!” command, and read the measurements with the “aD0!” command. This process is repeated, progressing through the value of “n”, until the “aMn!” command responds with less than 9 measurements.

When parameters are read back over the SDI-12 bus via one of the “aDn!” commands they will be reported in a fixed order (Table 19-2). If a unit has duplicate transducers, only one will be reported under this fixed-order reporting, by order of the port it is plugged into. For example, if two pH sensors are plugged into ports 1 and 3, the sensor in port 1 will be reported.

Note that the fixed reporting order can be changed as described in the tip ✔ on this page.

A measurement is returned for all possible MP TROLL parameters.

| Table 19-2. SDI-12 Parameter Reporting (Default Order) |
|---|---|---|
| Order | Parameter | Unit |
| 1 | Pressure | PSI |
| 2 | Temperature | degrees Celsius |
| 3 | Barometric Pressure | PSI |
| 4 | Turbidity | NTU |
| 5 | pH | pH |
| 6 | ORP | milliVolts |
| 7 | Conductivity (Actual) | µS/cm |
| 8 | D.O. (Polarographic) | mg/L |
| 9 | Battery Voltage | volts |
| 10 | Nitrate | ppm |
| 11 | Ammonium | ppm |
| 12 | Chloride | ppm |
| 13 | RDO (Optical D.O.) | mg/L |
| 14 | Conductivity (Specific) | µS/cm |
| 15 | Salinity | PSU |
| 16 | D.O. (Polarographic) | % saturation |
| 17 | RDO (Optical D.O.) | % saturation |
| 18 | Ammonia | ppm |
| 19 | Total Dissolved Solids | g/L |
| 20 | Resistivity | kOhm-cm |
• If a sensor port is empty, the unit will report readings with values of 0.0.

• If you edit the device to set SDI-12 mode preferences, as described in the tip ✔ above, only the parameters you select will be reported.

• If a sensor is reading out of its range, a value of -999999.9 will be reported.

REDUNDANT LOGGING (WIN-SITU 4 AND SDI-12)

If your MP TROLL 9500 supports internal logging, the instrument is capable of running tests (programmed in Win-Situ) while participating in an SDI-12 network; however Win-Situ cannot communicate with the MP TROLL 9500 while it is transmitting SDI-12 data, and conversely, the instrument cannot receive or respond to SDI-12 commands while connected to a PC serial port.

This “redundant logging” feature means

• if the SDI-12 recorder somehow “loses” data, the MP TROLL 9500 data can be retrieved using Win-Situ.

* if the SDI-12 recorder ceases to function due to power loss, the MP TROLL 9500 will continue to collect new data using its own internal batteries and clock.

TIP: Depending on the SDI-12 data recorder used, rapid sample schedules during a test may result in SDI-12 “retries.”

EXTENDED (ISCO) COMMANDS

Extended commands allow the data sampler to identify both the data channels and channel units that correspond to the ASCII formatted floating point data being returned. In response to the "aXPR0!" command, the response will be in the form: “aIxIxIx…<CR><LF>” where ‘a’ is the address, each ‘I’ is a parameter identifier, and each ‘x’ specifies the units for the preceding ‘I’.

The data sampler will first issue either a measure or a concurrent measure command. These commands return the number of channels that will be reported in a subsequent read command. This number will correspond with the number of pairs returned by the extended commands.

If the expected number of “Ix” pairs is not returned in response to the “aXPR0!” command, additional “aXPRx!” commands will be issued until all pairs are received.

REFERENCE

## SDI-12 V 1.3 COMMAND SET

<table>
<thead>
<tr>
<th>NAME</th>
<th>COMMAND</th>
<th>RESPONSE &amp; COMMENTS</th>
</tr>
</thead>
<tbody>
<tr>
<td>Address Query</td>
<td>?!</td>
<td>a&lt;CR&gt;&lt;LF&gt;</td>
</tr>
<tr>
<td></td>
<td></td>
<td>The wildcard address '?' character is supported only for the Address Query command. It is ignored as an invalid address for all other commands.</td>
</tr>
<tr>
<td>Acknowledge Active</td>
<td>a!</td>
<td>a&lt;CR&gt;&lt;LF&gt;</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Basic address characters in the range '0' to '9' and extended address characters in the ranges 'A' to 'Z' and 'a' to 'z' are supported. All other characters are ignored as an invalid address. The default address is '0'.</td>
</tr>
<tr>
<td>Change Address</td>
<td>aAb!</td>
<td>b&lt;CR&gt;&lt;LF&gt;</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Software changeable addresses and the Change Address command are supported.</td>
</tr>
<tr>
<td>Send Identification</td>
<td>all</td>
<td>013IN-SITU WQP100vvxxxxx&lt;CR&gt;&lt;LF&gt;</td>
</tr>
<tr>
<td></td>
<td></td>
<td>where vvv = device firmware * 100 (153 = 1.53) xxxxx = 5-digit device serial number</td>
</tr>
<tr>
<td>Start Verification*</td>
<td>aV!</td>
<td>0001&lt;CR&gt;&lt;LF&gt;</td>
</tr>
<tr>
<td></td>
<td></td>
<td>One result is available immediately for reading by the Send Data command.</td>
</tr>
<tr>
<td>Send Data</td>
<td>aD0! ... aD9!</td>
<td>0+0.0452<em>30+1.53+3.0+5</em>3.0+4.5&lt;CR&gt;&lt;LF&gt;</td>
</tr>
<tr>
<td>Start Measurement*</td>
<td>aM!</td>
<td>0001n&lt;CR&gt;&lt;LF&gt;</td>
</tr>
<tr>
<td></td>
<td></td>
<td>n parameters will be available for reading by the Send Data command within 1 second. A service request (a&lt;CR&gt;&lt;LF&gt;) will be sent when the parameters are ready. The number of parameters returned is determined by the SDI-12 defaults shown in Table 19-2 (or edit the SDI-12 mode preferences).</td>
</tr>
<tr>
<td>Send Data</td>
<td>aD0! ... aD9!</td>
<td>a-values&lt;CR&gt;&lt;LF&gt;</td>
</tr>
<tr>
<td></td>
<td></td>
<td>or a-values&lt;CRC&gt;&lt;CR&gt;&lt;LF&gt;</td>
</tr>
<tr>
<td>Additional Measurements*</td>
<td>aM1! ... aM9!</td>
<td>atttn&lt;CR&gt;&lt;LF&gt;</td>
</tr>
<tr>
<td>Additional Measurements with CRC</td>
<td>aMC1! ... aMC9!</td>
<td>atttn&lt;CR&gt;&lt;LF&gt;</td>
</tr>
<tr>
<td>Send Data</td>
<td>aD0! ... aD9!</td>
<td>a-values&lt;CR&gt;&lt;LF&gt;</td>
</tr>
<tr>
<td></td>
<td></td>
<td>or a-values&lt;CRC&gt;&lt;CR&gt;&lt;LF&gt;</td>
</tr>
<tr>
<td>Start Concurrent Measurement</td>
<td>aC!</td>
<td>atttnn&lt;CR&gt;&lt;LF&gt;</td>
</tr>
<tr>
<td>Start Concurrent Measurement with CRC</td>
<td>aCC!</td>
<td>atttnnn&lt;CR&gt;&lt;LF&gt;</td>
</tr>
<tr>
<td>Send Data</td>
<td>aD0! ... aD9!</td>
<td>a-values&lt;CR&gt;&lt;LF&gt;</td>
</tr>
<tr>
<td></td>
<td></td>
<td>or a-values&lt;CRC&gt;&lt;CR&gt;&lt;LF&gt;</td>
</tr>
<tr>
<td>Additional Concurrent Measurements</td>
<td>aC1! ... aC9!</td>
<td>atttnn&lt;CR&gt;&lt;LF&gt;</td>
</tr>
<tr>
<td>Additional Concurrent Measurements with CRC</td>
<td>aCC1! ... aCC9!</td>
<td>atttnnn&lt;CR&gt;&lt;LF&gt;</td>
</tr>
</tbody>
</table>

### Extended Commands

**ISCO Compatability**

aXPR0! ... aXPR9!<CR><LF>

where each Ix is a character pair identifying the parameter and units for each measurement. The number of Ix pairs equals the number of data values returned for the Start Measurement and Start Concurrent commands, limited to 19 per command. If the expected number of "Ix" pairs is not returned in response to the "aXPR0!" command, additional "aXPRx!" commands will be issued until all pairs are received.

---

* This command may result in a service request.

a Sensor address  ! Command terminator  <CR><LF> Response terminator

ttt Time (seconds) until measurement is ready  n, nn Number of measurement values
SDI-12 Schematic Installation Diagram

SDI-12 Data Recorder (customer-supplied)

up to 200 ft (61 m)

Adapter

to another SDI-12 device

up to 300 ft (91 m) Rugged Cable; see Table 19-1 for power requirements of cables longer than 170 ft (52 m)

MP TROLL 9500
Groundwater that has sat in a well casing for a period of time may not adequately represent the formation water. Well purging can ensure that fresh formation water is drawn into the casing for representative measurement of water temperature, pH, conductivity, and other parameters. The well may be pumped to remove a predetermined number of volumes of stagnant water from the well.

A more cost- and time-effective method of purging involves pumping at very low flow rates with concurrent monitoring of water quality indicator parameters until it is determined that true formation water is being monitored. The Flow-Sense Wizard in Win-Situ 4 and Pocket-Situ 4 automates this process to help determine when a representative sample of formation water can be collected from a well for analysis. Based on user input, the software calculates the volume of the In-Situ flow cell and tubing, and the time to exchange one complete volume at the specified pumping rate. The frequency of measuring the water-quality indicator parameters is based on the time required to completely evacuate one volume of the flow cell and tubing. Meter and graphical views help to determine when the change in parameter readings falls within the specified target range.

**PREPARATION**

- Install Win-Situ 4 from the software CD or In-Situ website
- Install Pocket-Situ 4 to your desktop/laptop PC from the CD or website, connect the computers in ActiveSync®, launch the Win-Situ Software Manager, and follow the instructions to install Pocket-Situ 4 on the RuggedReader
  
  **TIP:** When using the RDO sensor for low-flow monitoring, be sure to install the most recent version of Win-Situ 4 and Pocket-Situ 4, available at www.in-situ.com.
- Calibrate the water-quality sensors
- Have the following site information ready to enter in the software when prompted by the Flow-Sense Wizard:
  - Well diameter and total depth
  - Screen length
  - Depth to water level, top of screen and placement of pump intake, referenced to a benchmark
  - Pump model & type
  - Tubing inner diameter
  - Tubing type
  - Tubing length

**RDO SENSOR PREPARATION**

Special preparation is in order if you plan to use the sub-4" RDO® optical dissolved oxygen sensor for low-flow monitoring. You will need:

- RDO Optical Dissolved Oxygen sensor
- Flow Cell for use with RDO sensor

The RDO-ready flow cell includes hardware fittings for setting up the flow cell and installing the RDO sensor.

**PREPARE THE FLOW CELL**

Connect valves and tubing to flow cell body. Attach spike or base plate. Insert the calibrated TROLL 9500 into the flow cell. Turn on the pump.
**SECTION 20: LOW-FLOW MONITORING**

**CONFIGURE THE FLOW-SENSE WIZARD IN THE OFFICE**

Text entry on a PDA in the field can be tedious. To simplify the task, we suggest you run the Flow-Sense Wizard using Win-Situ 4 on a full-sized PC in the office to prime the software with representative values. Then use Win-Situ Sync to copy the “templates” created in this way to the PDA.

A. Connect the TROLL 9500 you will use in the field to your desktop PC. The device can be in air since the sampled data are not important.

B. For each well you need to sample, execute the Wizard as described on the following pages. The more information you can enter in advance, the less you will have to tap in later with a stylus outdoors.

C. Estimate any values you do not yet know (e.g., tubing length, pumping rate, final drawdown).

D. Let the stabilization phase run for at least one reading, then Accept.

E. When the Wizard asks if you want to save the file, give each one a meaningful name, for example:

   - “Well A template.flo”
   - “Well B template.flo”

F. On your next ActiveSync connection, In-Situ’s synchronization utility Win-Situ Sync will prompt you for Low Flow templates you wish to transfer to the PDA.

   **TIP:** If Win-Situ Sync does not launch automatically, select it from the In-Situ Inc. group in Programs on the Windows Start Menu. Be sure the option Transfer data files from Desktop to Field Unit is checked. Click to transfer files.

**START THE SOFTWARE**

Connect the TROLL 9500 to the PC or PDA. Launch Win-Situ 4 or Pocket-Situ 4. If you have not used the software before, take a moment to specify a connection type, COM port, and baud rate in the Connection Wizard.

**LAUNCH THE FLOW-SENSE WIZARD**

1. Select one of the following ways to launch the Flow-Sense Wizard:

   - Tools Menu: Select the Flow-Sense Wizard (not available in Pocket-Situ)
   - Navigation Tree: (a) Select the Flow-Sense Wizard in the Navigation tree; (b) Click or tap Start in the Information pane.

   - If you’ve used the Flow-Sense Wizard before—or copied “template” files from a different PC—expand the Flow-Sense Wizard folder by clicking on the +, then expand the Flow-Sense Data folder. (a) Select a file or template. (b) Click or tap Start.

Input values from that file or template will be copied.

**VERIFY UNIT PREFERENCES**

Before the Flow-Sense Wizard starts, you may wish to verify the current unit selections. Length measurements in the Flow-Sense Wizard default to metric (meters, centimeters), but you can enter and display this data in English units (feet, inches) if you prefer. You may also wish to verify the units for Conductivity, Dissolved Oxygen, ORP, pH, Temperature, and Turbidity.

   **TIP:** Flow units in the Flow-Sense Wizard will always be in milliliters (mL). For best results, units should be consistent between the desktop application and the PDA application.

To skip the settings verification, click Continue.

The Wizard will open the COM port and connect to the instrument. If you have used the Wizard before, or launched the Wizard from an existing .flo file, data entry values will be supplied.

   **TIP:** The Wizard opens the COM port you used last (this is stored in the Wizards.ini file in the Config subfolder in the folder where Win-Situ is installed).

   If several connections have been used in the past, the Wizard uses the first “direct” connection it encounters in the tree.

   If the Wizard is started without any connection information at all, it will attempt to connect using COM 1 at a baud rate of 19200.
FLOW-SENSE WIZARD INPUT

1. The first input screen provides for entry of specific details about the project. This information will be included in the output report. The Project Name and Site Name will appear in the output file name.

Click or tap Next to continue.

2. In the next screen, enter information about the well. This information will be included in the output report. The Well ID will appear in the output file name.

Click or tap Next to continue.

3. In the next screen, enter details about the pump and tubing (tubing information is used in later volume calculations). If creating a template, estimate the tubing length and pump placement.

Click or tap Next to continue.

The next screen requests pumping information

4. Final pumping rate: Enter in milliliters per minute. If creating a template, accept the default or estimate the pumping rate.

5. Total volume—Auto Calculated Value: This is the software-calculated volume of the cup (flow cell) and tubing, less the displacement of installed sensors.

To specify a different volume, clear the “Auto” check box, and enter the new volume (mL).
6. **Measurement interval—Auto Calculated Value**: This is the software-calculated time (seconds) for one complete exchange of water in the calculated volume at the final pumping rate. In other words, this is the time required to completely evacuate one volume of the flow cell and tubing and draw in a new volume. Readings will be taken at this interval unless you specify a different interval.

To specify a different interval, clear the “Auto” check box as shown above, and enter the desired measurement interval (seconds).

7. **Stabilized drawdown**: Final drawdown from the initial water level entered previously (measured with tape after pumping starts). If creating a template, estimate the final drawdown.

Click or tap **Next** to continue.

8. The final screen displays the available parameters. Choose the parameters to monitor and the target stability range for each parameter. You can enter absolute values in parameter units or percentage values.

9. If you wish to add comments, click the **Notes** button. They will be included in the output report.

10. Ensure the pump is on (unless you are creating a template), then click or tap **Start** to begin the stabilization readings.

The sidebar on page 139 describes the stabilization readings screen that is shown next.

11. When the readings appear stable, you are ready to save the data and exit the software. Click or tap **Accept**.

If creating a template, let the stabilization phase run for at least one reading, then click **Accept**.

**SAVING THE LOW-FLOW DATA OR TEMPLATE**

Click Save when a suggested output file name is displayed. You may change this name if you like. After it is saved, the file will be displayed in the Data Folder branch of the Navigation tree and will open in the Information pane, like other test files extracted in Win-Situ 4 or Pocket-Situ 4.

The file may be displayed in report format or as a graph in both Win-Situ 4 and Pocket-Situ 4. (If you can't see much on the PDA screen, try scrolling to the right.) The top of the file presents well, site, and project information. At the end of the file are the measurement data.

Additional functions are available through Win-Situ 4.

- Print (file menu)
- Graph (button in Information pane). However, note that better graphical report output is available if you export to Excel.
- Save as text file (file menu)
- Export to Excel (file menu)
OUTPUT

Low-Flow files are saved as files of type .flo in a folder named Flow-Sense Data under the Flow-Sense Wizard folder, and are accessible in the Navigation tree. By default they are named as shown below:

```
Flow-Sense Data \ Central Valley-New West Well-1562-6-10-2007.flo
```

Project name

Site name

Well ID

Date (m-dd-yyyy)

Low flow file designator

EXPORT TO EXCEL OPTION

To automatically create an output report in Microsoft® Excel® from Win-Situ 4:

1. In the Win-Situ Navigation tree, select a low-flow data file. If it opens in Graph view, select the Report button.
2. On the Win-Situ File Menu, select Export to Excel.
3. The report will open in an Excel spreadsheet.

USING A CUSTOM EXCEL TEMPLATE

The Win-Situ 4 installation includes an Excel template for creating an output report (shown on the following page). This template is named InSituLowFlow.xlt, and is automatically installed to the Templates directory of the Microsoft operating system. Brief instructions for using this template are included in the spreadsheet that opens when you select Export to Excel.

To format an output report using the custom template,

1. After exporting a low flow data file to Excel, insert a new sheet based on a template by right-clicking the tab at the bottom of the screen in Excel.
2. Click Insert, then select the template InSituLowFlow.xlt.
3. When prompted by Excel, select “Enable Macros.”

An example of low flow data formatted in Excel using this template is shown on the following page.

4. If desired, save the report as an .xls file.

STABILIZATION READINGS—METER AND GRAPH

To view this data in graphical form, click Graph.

The graph shows the change and stabilization ranges of all parameters. The “change band,” between the heavy dotted lines, represents a composite target of all specified stability ranges. You can drag the range finder up or down, or expand the stability region to zoom in.

The most recent readings are shown on the right, earlier readings on the left. 0 marks the latest reading. The change band is centered on the third point as a reference. The graph can show up to 10 sets of readings. Change is recalculated with each reading.

• Reading: the current reading
• Change - Val: difference between the maximum and minimum over the last 3 readings OR
• Change - %: difference between the current reading and the third reading back, expressed as a percentage
• Target specified earlier in the Wizard (shown for Values)
Sample of a Low-Flow output report using the Export to Excel function and the In-Situ template InSituLowFlow. 

*Normalized data = change in indicator parameters mapped from 0 to 1. A graph may be generated using Win-Situ 4 and inserted manually into the report, if desired.*
21 CARE & MAINTENANCE

REPLACING BATTERIES

The MP TROLL 9500 uses—

- two standard 1.5V alkaline D cells, or
- two 3.6V lithium D cells—recommended for use with an RDO optical dissolved oxygen sensor, and with a turbidity wiper

Use only Saft LSH-20 3.6V lithium D cells. Use of any other lithium battery will void the product warranty.

Battery voltage and approximate percentage remaining is displayed in the software interface when the instrument is connected to a PC.

Note: Due to the voltage supplied by two lithium D cells, the software may report that the TROLL 9500 is operating on external power.

To replace batteries:

1. Unscrew and remove the white battery compartment cover. If the cable is attached, slide the battery compartment cover up onto the cable.

2. Press down slightly on the top battery to remove it, or knock it out gently into your hand.

3. Tip the unit to slide the bottom battery out.

4. Insert the new batteries according to the diagram on the inside of the battery compartment (positive up for both).

5. Slide the white cover back down over the battery compartment and hand-tighten to thread it to the instrument body.

   Insert batteries negative side first, positive side up

   Screw the cover down firmly to compress the o-rings and create a waterproof seal. When properly assembled, the o-rings will not be visible.

6. At your next software connection in Win-Situ 4 or Pocket-Situ 4, edit the device to update the battery information. (See “Editing the Device Properties” in Section 4.)

O-RING SEALS

LUBRICATION

The Viton® o-rings used in the MP TROLL 9500 and other submersible In-Situ instruments are crucial to insure the integrity of the water-tight seal. We recommend that you inspect them each time they are stressed (insertion/removal of sensors, attachment/removal of the restrictor, battery replacement, etc.) for any indication of dirt, cracks, tears, splitting, shredding, desiccation, and other damage. If the o-rings are in good condition, apply silicone lubricant before re-assembling the instrument. Remove excess lubricant with a lint-free tissue. If the o-rings are damaged, they should be replaced, as described below.

When lubricating the sensor o-rings, take special care to keep grease away from the area around the connector at the bottom of the sensors. Should lubricant get into this area, remove it with a clean cotton swab.
REPLACEMENT

If the o-rings become damaged to the extent that no longer provide an effective seal, they should be replaced. If there is any doubt whether the o-rings should be replaced, it is best to err on the side of safety and replace them.

Before replacing o-rings, clean all mating surfaces, including the o-ring grooves.

O-rings and lubricant are included in the MP TROLL 9000 Maintenance kit available from In-Situ Inc. or your distributor.

GENERAL CLEANING

Rinse the instrument body well, especially if it has been in contact with contaminated media. Air-dry or wipe with a lint-free tissue.

Ultrasonic cleaning is not recommended.

STORAGE

Store the TROLL 9500 clean and dry. Place the protective red dustcap on the cable end, or store with cable attached to protect the connector pins and o-ring.

Store the instrument where it will be safe from mechanical shocks that may occur, such as rolling off a bench onto a hard surface.

Protect the instrument from temperature extremes. Store within a temperature range of -40°C to +80°C (-40°F to +176°F).

If the sensors are removed for storage, place plugs in the dry sensor ports as protection from dust and dirt.

SENSOR STORAGE

For long-term storage, return the water-quality sensors to their original packaging. Protect the lubricated o-rings from dust and dirt.

For up to a week, the sensors may be left in the instrument, with a moist sponge in the bottom of the Cal Cup to provide a moist environment for those sensors that require it.

TWIST-LOCK CONNECTORS

Keep the pins on all connectors free of dirt and moisture by using the soft protective dustcap when cable is not attached.
TROUBLESHOOTING CONNECTIONS

Problem: Win-Situ or Pocket-Situ cannot “find” (connect to) the MP TROLL 9500. Error 6146 may be displayed.

Probable Cause: Wrong COM port selected, loose cable connections, device is taking a measurement as part of a test, batteries are low, elastomer is worn

Suggested Remedy: Check the following:
- all cable connections are tight
- the back end is securely attached to the instrument
- the correct COM port is selected
- the internal battery has voltage remaining; attach external power
- If a test is running, try connecting again—several times if necessary to “sneak” in between test data points.

TROUBLESHOOTING DATA COLLECTION (TESTS)

Problem: Test ABENDed (came to an “ABnormal END”)
Probable Cause: Device lost power or ran out of memory
Suggested Remedy: None; indication of ABEND in software cannot be reversed, but data collected up until the time the test ABENDed is likely to be fine
- Check clock, check memory free, check device power

Problem: Scheduled test did not start
Probable Cause: Incorrect device clock, full memory, power removed at time of first scheduled data point
Suggested Remedy: Synchronize device clock and reschedule test; insure device has sufficient battery power and free memory; insure device is powered at time of first scheduled data point
TROUBLESHOOTING SENSORS

Problem: Sensor will not go into port
Probable Cause: (1) Insufficient lubrication on sensor o-rings. (2) Incorrect sensor alignment.
Suggested Remedy: (1) Generously lubricate sensor o-rings with a good silicone lubricant. Remove excess lubricant with a tissue, and take care to keep grease away from the area around the connector at the bottom of the sensor. (2) Align the mark on the side of the sensor (it looks like a small white “V”) with the tic mark on the sensor port. If you have trouble locating these marks, visualize the sensor block as a clock face, with the pressure/turbidity sensor or plug at 12:00. The port alignment marks are at the 3:00 position for each port.

Problem: Sensor will not come out of port
Probable Cause: Inability to grasp sensor
Suggested Remedy: Try the sensor removal tool. Insert it between the widest part of the sensor and the instrument body and press down on the handle, prying the sensor up until it pops out.

Problem: Software does not recognize sensor in port
Probable Cause: (1) Sensor is not firmly seated in port. (2) Excess lubricant or dirt in port. (3) Sensor is in a wrong port for its type.
Suggested Remedy: (1) Re-insert the sensor: Align the mark on the side of the sensor (it looks like a small white “V”) with the tic mark on the sensor port. Use sensor insertion tool to press sensor firmly into port until you feel it dock with the connector. When properly inserted a only a very small gap (0.060-0.075 inch, the width of the sensor removal tool) remains between the widest part of the sensor and the instrument body. (2) Remove excess lubricant and/or dirt from the connector on the sensor and from the connector in the port. (3) Insure the sensor is in the correct port for its type. Some sensors will function properly in any port, others will work only in specific ports. Refer to diagrams in section 3 or specific sensor section of this manual. Remove sensor and re-install in correct port, if necessary, then refresh device information in the software.

TROUBLESHOOTING CALIBRATION

Problem: The Cal Cup leaks when rinsing sensors
Probable Cause: Cal Cup is not tightened to probe body sufficiently to seat against the o-ring
Suggested Remedy: Carefully align threads on Cal Cup with threads on instrument body. Thread the Cal Cup onto the body until it is seated against the o-ring, then back off slightly to avoid overtightening.

Problem: The DO readings stabilized during the Quick Cal but now the readings are off (too high or too low)
Probable Cause: (1) Too much solution in Cal Cup during the Quick Cal to expose the DO sensor to air. (2) Cal Cup is too tightly sealed during calibration.
Suggested Remedy: (1) The DO membrane must be exposed to air for a valid 100% DO calibration. If the membrane is submerged when you invert the Cal Cup during the Quick Cal, remove the end cap and pour out some of the calibration solution until the membrane is in air. (2) Loosen the end cap of the inverted Cal Cup during DO calibration to avoid pressurizing the chamber.
APPENDIX

ELECTRONIC DRIFT AND DEVICE RECALIBRATION

The electronics of the Multi-Parameter TROLL 9500 will experience accuracy drift over time. This drift applies to all channels and is additive to the initial calibration accuracy. Electronic drift does not apply to user-calibrated sensors because the drift is compensated for during calibration.

The system wide long-term drift is dominated by the stability of the voltage reference used in the device. Other components exhibit long-term drift of a much smaller magnitude.

The equation used to calculate drift over time is:

\[ D = K \times \sqrt{\frac{t}{1000 \text{ hours}}} \]

where:

- \( D \) is the drift in ppm
- \( K \) is the long term stability coefficient listed as ppm / 1000 hours but actual units are ppm
- \( t \) is the time expressed in hours

Using this information, the maximum drift over time is shown in the table on this page.

<table>
<thead>
<tr>
<th>Time Period</th>
<th>Drift</th>
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<tr>
<td>Years</td>
<td>1K Hours</td>
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<tr>
<td>0.00</td>
<td>0.00</td>
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<tr>
<td>0.25</td>
<td>2.19</td>
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<tr>
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<tr>
<td>0.75</td>
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<td>8.76</td>
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<tr>
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<tr>
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<td>41.61</td>
</tr>
<tr>
<td>5.00</td>
<td>43.80</td>
</tr>
</tbody>
</table>

TIP: The recommended frequency of factory recalibration of the MP TROLL 9500 depends upon the amount of drift a user is willing to tolerate. For example, if a drift of 0.025% is acceptable, then the recalibration period is 6 months; if a drift of 0.05% is acceptable, then the recalibration period is 2 years.

DETERMINING DENSITY

The density of water in a well can be determined using an accurate tape measure and a pressure transducer. A change in pressure is recorded between two points as the transducer is lowered into the water and the corresponding change in depth is recorded using the measuring tape. Density is then calculated using a simple formula. This method is valid if the pressure and depth measurements are accurate and the water within the well is homogeneous throughout the entire depth of the well.
Note: Density can also be measured using a hydrometer if it is possible to withdraw a water sample from the well. We recommend an accuracy of ±0.0005.

Procedure

There are two possible methods for measuring the change in water depth. Choose the method that is the most convenient and accurate.

- Attach the tape measure to the transducer cable using an adhesive (e.g., duct tape). The tape measure can be attached to the transducer itself or to a segment of the cable that will be adjacent to the top of the well casing. Read the cable positions directly from the measuring tape. The measuring device must have a resolution of at least 1 mm or 1/16 in.

- Alternatively, mark the positions of the cable with an indelible felt tip pen. The distance between marks is then determined with a tape measure after the corresponding cable segment is removed from the well.

1. Lower the transducer into the well until it is submerged under about one meter of water.

2. Secure the transducer at a fixed depth using the cable at the top of the well casing. Wait an hour or so for the system to equilibrate.

3. Take an electronic pressure reading, manually, from the transducer and record this measurement in PSI. This is measurement P₁.

4. Read the tape measure relative to some fixed reference point (e.g., top of well casing) or mark the position on the cable with an indelible pen. This is measurement L₁.

5. Lower the transducer at least three meters deeper into the water and repeat steps 3 and 4. These are measurements P₂ and L₂.

6. Density (ρ) in g/cm³ is calculated using the following:

\[ \rho = \frac{(P_2 - P_1) \times 6.894757}{g \times (L_2 - L_1)} \]

where g is the gravitational acceleration for the location of the well in m/s². P must be in PSI units and L must be in meters. If using a tape measure calibrated in feet, 1 ft = 0.3048 m (exactly).

An error of 0.001 m (1 mm) in the depth measurement translates into an error of 0.00085 g/cm³ for density.
Glossary

ABend: Indication in the software interface that a test has come to an “ABnormal END”—usually because the device memory is full, or power was lost.

ABS: Acrylonitrile Butadiene Styrene, a plastic material.

Absolute pressure sensor: Non-vented pressure sensor that measures all pressure forces detected by the strain gauge, including atmospheric pressure. Fluid levels measured with an absolute pressure sensor must be corrected through subtraction of the atmospheric pressure to obtain accurate fluid level measurements. Compare Gauged pressure sensor.

AC: Absolute or Actual Conductivity.

Accuracy: Closeness of agreement between the result of a measurement and the true value. Usually expressed as a deviation from 100% accuracy.

A/D (analog to digital) converter: Converts an electrical signal to a numeric value that can be interpreted by a computer.

Ambient pressure (temperature): The pressure (temperature) of the medium surrounding the sensor or instrument.

Ammonia (NH₃): A toxic, colorless gas with a pungent odor, highly water-soluble.

Ammonium (NH₄⁺): Solvated ammonium cation produced when ammonia gas is dissolved in water.

Ammonium chloride (NH₄Cl): A salt used to make ammonium and chloride calibration standards.

Anion: Negatively charged ion (e.g., Cl⁻, NO₃⁻).

Atmospheric pressure: Pressure due to the atmosphere, altitude-dependent.

Barometric pressure, see Atmospheric pressure

Bithermal: At two temperatures, e.g., a three-point calibration of an ammonium, chloride, or nitrate sensor conducted at two different temperatures.

Basic sensor set: pH, pH/ORP, conductivity, and polarographic (Clark cell) dissolved oxygen; compare Extended sensor set.

Boxcar filtering: A smoothing scheme that looks at the 5 most recent data points.

Cable, see Communication cable, Network cable, Rugged Cable, TROLL Com, Vented cable.

Calibration: The process of determining the response of a measurement system to a known amount of the measured component in order to permit the measurement of unknown samples.

Calibration, out-of-box: Use of new Basic sensors for the MP TROLL 9500 directly from the box, with factory-supplied calibration coefficients. Sensors should be calibrated after they are used and begin to drift.

Calibration, Quick Cal: A rapid calibration procedure available for the Basic sensors, using a single solution.

Calibration, traditional: Single- or multi-point methods for calibrating water-quality sensors individually to gain higher accuracy.

Calibration coefficients: Sensor slope and offset that convert analog measurements to user units; calculated during calibration.

Calibration cup: Clear PVC vessel that attaches to the front end of the MP TROLL 9500 in place of the restrictor and holds the recommended amount of calibration solution during calibration of the water-quality sensors.

Calibration kit: Boxed set of standards for calibrating a specific water-quality sensor for operation in a specific range.

Calibration solution, Calibration standard: A solution whose concentration is accurately known.
Cation: Positively charged ion (e.g., NH₄⁺, Na⁺).

Cell constant: A value that describes a conductivity cell which is determined by electrode area and electrode separation.

Chloride (Cl⁻): Common anion in water that gives it a salty taste.

Chlorophyll: Green pigment in plants that transforms light energy into chemical energy during photosynthesis.

Clone: Copy all elements of a selected test definition to a new test.

Combination electrode: A combination of a sensing or measurement electrode and a reference electrode in one unit.

COM port: RS232 serial communication port on a PC.

Communication cable: see TROLL Com

Conductivity, electrical: A measure of the ability of an aqueous solution to carry an electric current. Increases with increasing temperature.

Connection node: Node in the Win-Situ or Pocket-Situ Navigation tree, representing the COM port on the host PC.

Data folder: Node in the Win-Situ or Pocket-Situ Navigation tree, providing a view of tests that have been extracted from the device memory to the host PC. The Data Folder can be expanded to show the device type, device serial number, and test name.

Data point: In logged data, one reading from every channel being sampled.

Density: Mass of a substance per unit volume (e.g., grams per liter, g/L; grams per cubic centimeter, g/cm³). Water is most dense at 4° Celsius.

Depth: The distance between the water surface and the pressure sensor of the MP TROLL 9500. Converted from pressure units using values selected by the user during pressure parameter setup.

Derived measurement: Value calculated in software from the output of more than one sensor; e.g., salinity is derived from conductivity and temperature.

Device node: Node in the Win-Situ or Pocket-Situ Navigation tree, representing the connected MP TROLL 9500.

Digital DO: Proprietary method of powering the dissolved oxygen sensor; digital pulsing eliminates the need to stir the sample to avoid oxygen depletion.

Dissolved oxygen (DO): The amount of oxygen present in water and available for respiration.

DO: Dissolved Oxygen.

Downhole cable, see RuggedCable

Drift: Gradual change in sensor response with time.

DSP: Digital signal processor.

Electrode: An electric conductor through which an electric current enters or leaves a medium (such as an electrolyte).

Electrolyte: A chemical compound which when dissolved in water will conduct an electric current; sensor filling solution.

Event test, Event sampling: Test measurement schedule in which the selected parameters are measured at the same regular, unvarying linear interval, but the data are logged only if the measurements on the designated event channel exceed a user-specified value. Conserves storage memory while logging all meaningful data.

Extended sensor set: Ammonium, chloride, nitrate, turbidity, and optical dissolved oxygen sensors; compare Basic sensor set.

Extract: Copy test data from the MP TROLL 9500 memory to a host PC. Initiated by the Extract button when a test is selected in the Navigation tree.

Firmware: Software program that resides in the memory of the MP TROLL 9500; firmware can be field-upgraded.

Flow cell: Clear vessel with input and output ports for routing flow past the sensors of the MP TROLL 9500; alternative to in-situ installation of the instrument where in-place installation is not possible or practical.

Flow-Sense Wizard: Low-flow application in Win-Situ software for monitoring water-quality indicator parameters in a flow cell during low-flow pumping.

FNU: Formazin Turbidity Units; compare NTU

FS: Full scale.

Gauged pressure sensor: Pressure sensor that is vented to the atmosphere; measures only pressure exerted by the water column, excludes the atmospheric pressure component. Compare Absolute pressure sensor; see also Vented cable.

HDPE: High-density polyethylene.


Home site: Default site node (top node) in the Win-Situ or Pocket-Situ Navigation tree, representing the host computer.
Host PC: Desktop computer running Win-Situ, or PDA running Pocket-Situ, connected to the MP TROLL 9500 via TROLL Com or other communication interface.

Interference: The presence of a species in a sample other than the species being measured that causes erroneous values.

Ionic strength: A measure of the total effect of all the ions, both positive and negative, present in a solution.

ISA (Ionic Strength Adjustor): Solution of high ionic strength which can be added to both sample and calibration solutions in equal proportions before measurement to minimize differences in ionic strength.

ISE (Ion-Selective Electrode): An electrode which responds selectively to the ions of a particular species in solution.

Isopotential point: The ion concentration at which changes in temperature do not cause a change in ISE sensor response (voltage).

Isothermal: At constant temperature.

Kcell, see Cell constant

KCl: Potassium chloride.

LED: Light-emitting diode, used in turbidity and Optical DO sensors.

Level reference: User-specified starting point for level readings; entered during pressure parameter setup.

Level—Surface: A mode for displaying logged pressure measurements; readings are positive up; useful for measuring surface water; permits use of zero or other user reference.

Level—Top of Casing: A mode for displaying logged pressure measurements; readings are positive down, as the water level draws down from the top of the well casing; permits entry of zero or other user reference.

Limnology: The science of the life and conditions for life in lakes, ponds, and streams.

Linear average: Test measurement schedule in which each measurement stored to the data file is the average of multiple closely-spaced measurements.

Linear test, Linear sampling: Test measurement schedule in which the selected parameters are measured at the same regular, unvarying sample interval and all measurements are logged.

Log test, Log sampling: Test measurement schedule in which measurements begin closely spaced and the interval between measurements continuously increases. Short for Logarithmic sampling.

LSZH: Low smoke zero halide

µM, µmol/L: micro Molar, unit of dissolved oxygen concentration; to convert to mg/L, divide by 31.25.

mg/L: Milligram per liter; equivalent to one part per million (ppm).

miniTROLL: In-Situ’s 0.72” diameter downhole pressure/temperature smart probe.

Molarity (M): A unit of measure indicating concentration in moles of solute per liter of solution.

Multi-ISE: An ion-selective electrode that measures more than one parameter.

NaNO₃: Sodium nitrate.

Na₂SO₃: Sodium sulfite.

Navigation tree: Left side of the Win-Situ application interface, or top of the screen in Pocket-Situ, showing selectable nodes: Site, Data Folder, Connection, Device, Parameters, Tests.

Nernst equation: The fundamental equation that relates the electrode potential to the activity of measured ions in a solution.

NH₄Cl: Ammonium chloride.

N.I.S.T.: National Institute of Standards and Technology, a non-regulatory federal agency within the U.S. Department of Commerce. Formerly known as the National Bureau of Standards.

Nitrate (NO₃⁻): Oxidized form of nitrogen that is highly soluble in water, present in soils, fertilizer, wastewater, etc.

Node: Element in the Navigation tree on the left side of the Win-Situ interface, or at the top of the screen in Pocket-Situ. When a node is selected, the remainder of the screen displays details about the node. Nodes include the Site, Data Folder, Connection, Device, Parameters (group node), single Parameter, Tests (group node), single Test.

Non-vented pressure sensor, see Absolute pressure sensor

Nose cone: Detachable threaded protective stainless steel piece at the front end of the MP TROLL 9500.

Nose cone stirrer, see Stirrer

NTU: Nephelometric turbidity unit, a measure of the intensity of light scattered by a water sample. From nephelometer, a type of turbidity meter.
dimeter. Comparable to previously reported Formazin Turbidity Unit (FTU), and Jackson Turbidity Unit (JTU).

Optode: optical electrode.

ORP, see Oxidation-reduction potential

Oxidation-reduction potential (ORP), also called “redox” potential (from “reducing” and “oxidizing”): Voltage difference at an inert electrode immersed in a reversible oxidation-reduction system; measurement of the state of oxidation of the system.

Parameter node: Node in the Win-Situ or Pocket-Situ Navigation tree, representing a single parameter (pressure, temperature, pH, conductivity, etc.).

Parameters node: Node in the Win-Situ or Pocket-Situ Navigation tree, providing a view of all parameters the device can measure.

Partial pressure: In a mixture of gases, the pressure a single gas would exert if it occupied the entire volume.

Pascal: Unit of pressure equal to the pressure resulting from a force of 1 newton acting uniformly over an area of 1 square meter.

PC: Desktop or laptop computer.

PDA (Personal Data Assistant): Generic term for a hand-held personal computer.

pH: Term used to describe the hydrogen-ion activity of a system; the negative logarithm of the activity of the hydrogen ions (H+) in the solution.

Pocket PC: A type of PDA with an ARM processor and Pocket PC (Windows Mobile) operating system.

Pocket-Situ: Win-Situ 4 software for supported PDAs.

PocketSync for Pocket-Situ: Synchronization utility that runs on a desktop/laptop PC; automatically installs or updates Pocket-Situ on a connected PDA; synchronizes data and other files between the PDA and the PC where Win-Situ is installed.

Polarization: Application of a direct or alternating current to a sensor.

Potassium chloride (KCl): A salt used to make conductivity calibration standards.

ppm: Part-per-million; equivalent to a milligram per liter (mg/L).

Precision: The closeness of agreement between independent test results obtained under stipulated conditions. A measure of the reproducibility of a method.

Pressure: A type of stress which is exerted uniformly in all directions. Its measure is the force exerted per unit area; e.g., pounds per square inch (psi), newtons per square meter (pascals).

Pressure transducer: Instrument or component that detects a fluid pressure and produces an electrical signal related to the pressure.

Professional: MP TROLL 9500 model containing memory; able to log data; accommodates the Basic sensor set.

Professional XP: MP TROLL 9500 model with all features of the Professional model and in addition allows use of the Extended sensor set.

Profiler: MP TROLL 9500 model without memory; must be used with a PC or PDA; accommodates the Basic sensor set.

Profiler XP: MP TROLL 9500 model with features of the Profiler and in addition allows use of the Extended sensor set.

Profiling: Taking continuous real-time readings of all enabled parameters.

PRT: Platinum resistance thermometer, a type of resistance temperature detector (RTD).

psia: A pressure unit, pounds per square inch absolute, measured with respect to zero pressure. All forces detected by the strain gauge are measured, including atmospheric pressure.

psig: A pressure unit, pounds per square inch gauge, measured with respect to atmospheric pressure. Thus the atmospheric pressure component is excluded.

PSU: Practical Salinity Units, based on the Practical Salinity Scale.

Pulsing: Periodic low-frequency polarization of a dissolved oxygen sensor.

Pump test, Pumping test: Aquifer characterization test that involves pumping out a known volume of fluid from a well and measuring the time of recovery to stable conditions.

Quick Cal: Rapid calibration procedure available for the MP TROLL 9500 Basic sensors that uses a single solution.

Quick Cal solution: A calibration standard of an appropriate chemical composition to calibrate four parameters simultaneously (pH, ORP, Dissolved Oxygen, Conductivity).

Quinhydrone: Dark green water-soluble compound, a 1:1 complex of benzoquinone and hydroquinone used as an ORP calibration standard when dissolved in pH buffer solution.
Reading: Measurement from a single channel (parameter).

Redox potential, see Oxidation-reduction potential

Reference (water level measurements), see Level reference

Reference electrode: A standard electrode of known potential against which the measurement or sensing electrode is compared.

Repeatability: Closeness of the agreement between the results of successive measurements carried out under the same conditions.

Reproducibility: Closeness of the agreement between the results of measurements of the same measurand carried out under nearly identical conditions but after an intermediate change was made and removed.

Resistance thermometer: Temperature sensor that changes electrical resistance with temperature.

Resistivity: The reciprocal of conductivity; calculated from conductivity.

Resolution: The smallest unit that can be measured by a device over its full range.

Response time: Time required for the MP TROLL to power a sensor and the sensor to return an accurate reading.

Restrictor: Perforated stainless steel area of the MP TROLL 9500 between the nose cone and the body; protects the sensors and allows free circulation of environmental fluid.

RDO: Rugged optical dissolved oxygen sensor

RS-485: Communications protocol using Recommended Standard 485 of the Electronic Industries Association (EIA-485) for the orderly transfer of electrical data signals; a balanced (differential), multipoint Interface Standard that uses two lines to transmit/receive data and can operate at 100 Kbps with cable lengths up to 4000 feet.

RTD: Resistance temperature detector, a type of resistance thermometer that has nominally 100 Ω at 0°C.

RuggedCable™: Waterproof, submersible, TPU-jacketed vented or non-vented cable for a Multi-Parameter TROLL 9500; carries power and communication signals; provides strain relief and a means to anchor the instrument to a stationary object; Twist-Lock connectors on both ends, Rugged (titanium) or Standard (carbon-filled ABS plastic); halogen-free version available.

S, see Siemens

Salinity: A measure of the amount of salts dissolved in water, usually expressed in parts per thousand (ppt); calculated from conductivity and temperature.

Saturation: The point at which a substance contains a maximum amount of another substance at a given temperature and pressure.

SC: Specific Conductance, conductivity corrected to 25°C.

SDI-12: A serial-digital interface operating at 1200 baud.

Sensor kit: Package containing an In-Situ sensor, along with necessary installation items and instructions for installing in the Multi-Parameter TROLL 9500.

SI: International System of Units.

Siemens (S): SI unit for conductivity, reciprocal of the ohm; this unit was formerly known as the “mho” (ohm spelled backwards).

Site node: Topmost node in the Win-Situ or Pocket-Situ Navigation tree, representing the host computer.

Slope: Sensor response vs. concentration (quantity). Slope and offset are the coefficients calculated during calibration that convert analog measurements to user units.

Slug test: Aquifer characterization test that involves “slugging” a well with a known volume of fluid or solid and measuring the time of recovery to stable conditions.

Soak time: Length of time a sensor is immersed in a calibration solution or sample, ideally at the same temperature at which measurements will be taken.

Sodium nitrate (NaNO₃): A salt used to make nitrate calibration standards.

Specific conductance: Conductivity of a solution at 25°C.


Stirrer: Battery-powered motorized low-power stirring mechanism with magnetic stirring bar for use during calibration and/or in stagnant waters.

Submersible cable: Waterproof, quick-connect cable designed for submersion; Instrument backshell located at downhole end; surface connector at surface end.
Surface connector: Multi-function vented nylon connector at the surface end of the MP TROLL’s submersible cable; accommodates a variety of top-of-well devices for communications, networking, and power supply.

TDS, see Total Dissolved Solids

Tefzel®: Dupont®’s modified ETFE fluoropolymer

Temperature, Solution temperature: Amount of heat present in the solution in which the instrument is submerged.

Test: Instructions to the MP TROLL’s internal logger for collecting data; the logged data from one set of instructions.

Test node: Node in the Win-Situ or Pocket-Situ Navigation tree, representing a single test.

Tests node: Node in the Win-Situ or Pocket-Situ Navigation tree, providing a view of all tests currently stored in the device memory.

TOC (Top of Casing), see Level—Top of Casing

Torr: A unit of pressure, equal to 1/760 atmosphere.

Total Dissolved Solids (TDS): The amount of dissolved substances, such as salts or minerals, in water remaining after evaporating the water and weighing the residue. Calculated from conductivity.

TPU: Thermoplastic Polyurethane, a cable jacket option.

TROLL Com: Communication cable interface between MP TROLL 9500 and a desktop/laptop PC or RuggedReader handheld PDA.

Turbidity: A measure of the transparency of water.

Twist-Lock Hanger: Back end hanger without venting or communication capabilities; allows use of inexpensive hanging cable while taking absolute (non-vented) pressure data with a preprogrammed instrument.

Units: Measurement units; user-selectable in software interface.

Vented cable: RuggedCable with a vent tube that applies reference atmospheric pressure to the back of the pressure sensor diaphragm.

Vented pressure sensor, see Gauged pressure sensor

Vertical profile: Characterization of a water column from surface to bottom (or vice-versa) through multiple real-time readings of the water-quality parameters of interest taken at varying depths.

Win-Situ 4: Instrument control software for instrument setup, calibration, profiling, data logging, data retrieval and display.

ZoBell’s: A redox standard solution with a known state of oxidation-reduction potential (measured in milliVolts) used to calibrate ORP.
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Declaration of Conformity

Manufacturer: In-Situ, Inc.
221 East Lincoln Avenue
Ft. Collins, Co. 80524
USA

Declares that the following product:

Product name: Multi-Parameter TROLL 9500
Model: WQP-100
Product Description: The Multi-Parameter Troll 9500 provides real-time readings for and logs up to 9 water-level and water-quality parameters 7 of which are multi-configurable in addition to the built-in temperature and barometric pressure parameters.

is in compliance with the following Directive

89/336/EEC for Electromagnetic Compatibility (EMC)

and meets or exceeds the following international requirements and compliance standards:

- **Immunity**
  EN 61326:1997, Electric Equipment for Measurement, Control and Laboratory Use

- **Emissions**
  Class A requirements of EN 61326:1997, Electric Equipment for Measurement, Control and Laboratory Use

Supplementary Information:
The device complies with the requirements of the EU Directive 89/336/EEC, and the CE mark is affixed accordingly.

Todd Campbell
New Product Development Program Manager
In-Situ, Inc.
October 28, 2005
Declaration of Conformity

Manufacturer: In-Situ, Inc.
221 East Lincoln Avenue
Fort Collins, CO 80524
USA

Declares that the following product:

Product name: TROLL Com
Model: USB TROLL Com
Product Description: RS485 to USB converter

is in compliance with the following Directive


and meets or exceeds the following international requirements and compliance standards:

- **Immunity**
  - EN 61326, Electrical Equipment for Measurement, Control and Laboratory Use, Industrial Location

- **Emissions**
  - Class A requirements of EN 61326, Electrical Equipment for Measurement, Control and Laboratory Use

Supplementary Information:
The device complies with the requirements of the EU Directives 89/336/EEC and 73/23/EEC, and the CE mark is affixed accordingly.

Todd Campbell
New Product Development Program Manager
In-Situ, Inc.
June 17, 2006
Appendix E

Sample Label

Chain of Custody
<table>
<thead>
<tr>
<th>Client:</th>
</tr>
</thead>
<tbody>
<tr>
<td>Project ID:</td>
</tr>
<tr>
<td>Sample ID:</td>
</tr>
<tr>
<td>Collection Date:</td>
</tr>
<tr>
<td>Time:</td>
</tr>
<tr>
<td>Tests:</td>
</tr>
</tbody>
</table>
# Chain of Custody Record

**GeoLabs, Inc. Environmental Laboratories**

45 Johnson Lane, Braintree, MA 02184  
P 781.848.7844  F 781.848.7811  
www.geolabs.com

**Sample Handling**
- **circle choice**
  - Filtration: Done  
  - Not Needed  
  - Lab to do  
- Preservation: Lab to do  Y/N

**Data Delivery**
- **circle choice (s)**
  - Fax  
  - Email  
  - Format: Excel  
  - PDF  
  - QC  
  - Other

**Requirements**
- **circle choice (s)**
  - GW-1  
  - MCP Methods  
  - S-1  
  - DEP  
  - CT RCP (Reasonable Confidence Protocols)  
  - State / Fed Program - Criteria

**Client Information**
- **Name:**
- **Address:**
- **Phone:**
- **Fax:**
- **Email:**

**Project Information**
- **Project:**
- **Project PO:**
- **Invoice to *:**

**Collection Details**

<table>
<thead>
<tr>
<th>DATE</th>
<th>TIME</th>
<th>SAMPLE LOCATION / ID</th>
<th>SAMPLE TYPE</th>
<th>QUANTITY</th>
<th>MATRIX</th>
<th>GRAB</th>
</tr>
</thead>
</table>

**GeoLabs SAMPLE NUMBER**

**Preservatives**

<table>
<thead>
<tr>
<th>1 = HCl</th>
<th>2 = HNO3</th>
<th>3 = H2SO4</th>
<th>4 = Na2SO3</th>
<th>5 = NaOH</th>
<th>6 = MEOH</th>
<th>7 = Other</th>
</tr>
</thead>
</table>

**Containers**

<table>
<thead>
<tr>
<th>A = Amber</th>
<th>B = Bag</th>
<th>O = Other</th>
</tr>
</thead>
<tbody>
<tr>
<td>G = Glass</td>
<td>P = Plastic</td>
<td>S = Summa</td>
</tr>
</tbody>
</table>

**Matrix Codes**

| GW = Ground Water | DW = Drinking Water | S = Soil | A = Air |
| WW = Waste Water  | SL = Sludge         | O = Oil  | OT = Other |

**Received on Ice**

**Preservatives**

**Containers**

**Relinquished by:**

**Received by:**

*Terms: Payment due within 30 days unless other arrangements are made. Past due balances subject to interest and collection cost. 
Note: Homeowners and Lawn Firms must pay when dropping off samples. We accept cash, check and credit cards.*

CT [FH-0148]  MA (MA - 015)  NH (2508)  NJ (MA-009)  NY (11795)  PA (88-03417)  RI (LA000252)
Appendix F

Laboratory QA/QC Procedures
GEOLABS, INC.

COMPREHENSIVE LABORATORY QUALITY ASSURANCE PLAN

Version 20, April 1, 2010

45 JOHNSON LANE
BRAINTREE, MA 02184
(781) 848-7844

Authorized Signatures:

Charles Morrow, Laboratory Director

David Kahler, President

Mark Perkins, Quality Assurance Manager
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1.0 INTRODUCTION

GeoLabs, Inc. is an environmental and analytical chemistry laboratory with a strong commitment to providing quality services for government, commercial and industrial clients with water, air, soil and hazardous material analytical needs. GeoLabs provides these services to national, regional and local clients and is committed to providing superior quality work. GeoLabs, Inc. is a corporation registered in the Commonwealth of Massachusetts. Corporate headquarters is located in a 12500 square foot facility in Braintree, MA, 10 miles south of Boston. GeoLabs is staffed by over 25 professionals, including Chemists, Environmental Scientists, and other technical specialists.

1.1 Statement of Policy

GeoLabs recognizes that our achievement of excellence in analytical services depends on the accuracy and precision of the data we provide to our clients. As a result, we have developed a Quality Assurance/Quality Control (QA/QC) program which ensures that standardized, proper protocols are followed and documented for each sample analyzed. As a NELAP certified laboratory we are committed to compliance with the NELAC standards.

1.2 QA/QC Considerations

The complex task of producing high-quality data can be grouped into several QA/QC considerations:

- Samples must remain undisturbed and representative of sampled conditions until they are analyzed.
- Care must be taken that samples are properly preserved.
- Proper analytical procedures must be followed.
- Analytical equipment must be in proper working order.
- Procedures to determine acceptability of QC data must be formalized.
- Raw data must be "reduced" to usable, comparable formats.
- Procedures for dealing with unusual results/circumstances must be in place.
- All of the above items must be documented appropriately.

With these considerations in mind, GeoLabs has developed specific, uniform procedures for every step of sample handling, analysis, data management and review. While adapted to our own internal needs, these procedures are consistent with the QA/QC requirements of government agencies.

By our definition, those procedures concerned with the accuracy and precision of each sample analyzed fall into the QC category. These are usually single procedures which are performed in conjunction with analysis that are used to quantify the success of analysis. In the GeoLabs plan these include:

- Instrument calibration criteria
- Reagent and standard preparation
- Replicate/spike/blank protocols
- Determination of detection limits

QC activities associated with the procedures above are individually tailored to each instrument and analytical method and are described in the Standard Operating Procedure for each method.

QA, on the other hand, is the composite of all activities involved with the production of valid information. Documentation, review and procedural updating are key elements of GeoLabs' plan which includes the following:
Method selection and updating
Chain of Custody
Sample log-in and identification
Sample storage and integrity
Analysis scheduling (to minimize sample holding time)
Documentation of sample preparation and analysis activities
Documentation of standard and reagent receipt and preparation
Calculation of results
Data review
Preparation of final reports
QA performance audits and system audits
Personnel training
Safety

Periodic reviews of the entire QA plan are performed at least once a year prior to the annual internal audit to ensure that appropriate QA procedures are established and initiated in a timely manner. Each employee has clearly defined QA/QC responsibilities while responsibility for QA plan updating and auditing of the QA system rests with GeoLabs' Quality Assurance Officer.

This document details all of the QA procedures followed by GeoLabs staff. Several other documents exist as supplements to this one. These documents include the Standard Operating Procedures specific to each method. The Standard Operating Procedures include detailed QC requirements for each analysis.

1.3 Intended Use of The Quality Assurance Plan

The intended use of the GeoLabs QA Plan is to maintain the quality of each analysis and to insure that the laboratory results are accurate and are presented in a clear and concise manner to our customers. It is understood and recognized that clients will often be using GeoLabs' analysis data for reporting purposes to the appropriate regulatory agency. The environmental assessment of a potential remediation site is based almost completely on the final results generated by a state certified laboratory; therefore, it is imperative to ensure that all analytical methods are conducted under controlled conditions. Controlled conditions can be maintained only by implementing and enforcing a formal QA/QC program that guarantees analytical data can withstand the most vigorous scrutiny.
2.0 Organization of the Laboratory

Laboratory services are organized into individual disciplines: inorganic/wet chemistry, volatile organics, semi-volatile organics, metals, TPH/pesticides/PCBs, EPH/VPH, and volatiles in air analyses. Each discipline is responsible for its work output and quality. The Laboratory Director oversees all laboratory operations including the enforcement of quality control procedures as well as solving any technical problems that may arise in the laboratory. Qualification and continuing education/training documentation for all employees is maintained by the Laboratory. This section describes the specific roles and responsibilities that accompany positions at GeoLabs.
2.1 Sample Custodian/Project Coordinator

- Reports to the Laboratory Director
- Inspects all coolers, samples, and Chains of Custody upon receipt.
- Confirms proper sample preservation of non-volatile aqueous samples by measuring and recording temperature and pH, as required.
- Corrects preservation where necessary and makes appropriate note on Chain of Custody
- Informs client of any abnormalities
- Brings any abnormalities or unfamiliar requirements/tests to the attention of the Lab Director
- Assigns unique sample number, and maintains the log-in data base.
- Labels sample containers with unique sample numbers.
- Maintains a thorough knowledge of sampling/ receipt protocol.
- Monitors work flow
- Assembles and reviews project data for completeness
- Prepares sample bottles for delivery to clients

2.2 Chemist

- Reports to the Laboratory Director
- Performs extractions, digestions, concentrations and analyses according to the approved methodology.
- Documents each step legibly, completely, and accurately.
- Retains responsibility for the analysis and/or preparation of applicable quality control samples.
- Responsible for the documentation and reporting of applicable quality control data for control charts and method detection limit studies.
- Clearly documents and reports all instances of failed quality control procedures associated with samples.
- Operates all equipment according to the specifications of the manufacturer and the approved methodology.
- Maintains a thorough understanding of the QA Plan, QC criteria, and accepted laboratory practices.

2.3 Instrument Analyst

- Reports to the Laboratory Director
- Maintains, calibrates and operates instruments.
- Performs sample analyses according to approved methodology.
- Documents each step completely, legibly, and accurately.
- Retains responsibility for the analysis and/or preparation of applicable quality control samples.
- Responsible for the documentation and reporting of applicable quality control data for control charts and method detection limit studies.
- Clearly documents and reports all instances of failed quality control procedures associated with samples.
- Operates all equipment according to the specifications of the manufacturer and the approved methodology.
- Performs necessary calculations.
- Reviews data for correctness
- Maintains a thorough understanding of the QA Plan, QC criteria and accepted laboratory practices.

2.4 System Manager/Office Manager

- Reports to the President
- Responsible for in-house computer maintenance and troubleshooting
2.5 Report Specialist
- Reports to the Laboratory Director
- Performs data review for completeness
- Requests corrections by laboratory if necessary
- Produces final deliverable reports
- Submits final deliverable reports to the Quality Assurance Manager for review.

2.6 Financial Manager
- Reports directly to the President
- Serves as company CFO
- Serves as company Human Resource Manager

2.7 Quality Assurance and Safety Manager
- Reports directly to the President
- Maintains reference material on quality assurance procedures and requirements.
- Prepares and updates the QA Plan, in-house SOPs, QC criteria and QC protocols in strict accordance with approved methodology and/or regulations
- Monitors laboratory compliance with the QA Plan
- Arranges for and conducts internal audits on the entire quality system annually
- Prepares yearly report to management on deficiencies in the quality system and monitors corrective action
- Recommends new QA program implementation when necessary
- Produces in-house warning and control limits from QC data supplied by chemists/analysts
- Serves as the laboratory point of contact for the exchange of QA/QC information
- Performs review of final deliverable packages before submittal to Lab Director
- Prepares and updates the Laboratory Chemical Hygiene Plan
- Provides initial employee training on GeoLabs safety policies and procedures
- Does not routinely analyze samples.

2.8 Laboratory Director
- Reports directly to the President
- Maintains a well-versed technical understanding of analytical methodology for the evaluation of laboratory operations
- Has responsibility for the administrative oversight and overall operation of the laboratory, including defining minimum qualifications, experience and skills necessary for all technical employees. Establishes training guidelines and policies for new personnel. Annually reviews all laboratory personnel to ensure continued technical proficiency.
- Works to ensure the implementation of the quality system
- Directs all aspects of laboratory operations to ensure the timely completion of all obligations
- Monitors the progress of sample analysis
- Assists both the client and laboratory in resolving any difficulties that may arise during a project
- Authorizes use of data which exceeds normal QC criteria, but due to extenuating circumstances retains validity
• Reviews all data generated by the laboratory
• Certifies the content of final deliverable packages prior to release with a dated signature
• Retains ultimate responsibility for the quality of data generated and reported by the laboratory

2.9 President
• Retains ultimate responsibility for the company
• Initiates new programs as desired
• Coordinates laboratory services directly with clients

2.10 Relationships

As GeoLabs is a small company many of its relationships are open and informal. All staff are aware of technical and quality issues in their areas and are free to suggest ways of improvement or to point out potential problems. A great degree of cross-training exists among the office/login staff and among the laboratory staff. As necessary, responsibilities are assumed by other members of the team.

2.11 Ethics Policy

GeoLabs realizes the importance of ethical behavior on the part of all its employees. GeoLabs utilizes training at the time of initial employment to ensure that its employees are aware of what is involved in ethical behavior with respect to the laboratory environment. Employees sign a "Statement of Understanding" indicating that their questions have been satisfactorily answered and that they understand the issues involved. Annual ethics and integrity training is required under NELAC and will be scheduled by the President at the beginning of the calendar year. Statement of Understanding is kept in each employee's personnel file.

Management is always available to answer questions concerning particular situations.

GEOLABS ETHICS POLICY

It is GeoLabs' responsibility to ensure training of personnel in their ethical and legal responsibilities. The responsibility for training and/or delegation of training responsibilities to someone with the knowledge and experience necessary shall rest with the Laboratory Director. It is GeoLabs' responsibility to insure that employees are free from any commercial, financial, and other undue pressures, which may affect the quality of their work. If such pressures are encountered by an employee, it is their responsibility to notify the Laboratory Director and/or Quality Assurance Manager. These issues are then brought to the attention of the President for action. All complaints will be discussed seriously and monitored by management to ensure compliance.

Below are the criteria that will be used by GeoLabs to determine what constitutes an improper, unethical, or illegal action.

Improper Actions: Improper actions are defined as deviations from GeoLabs standard operating procedure specified analytical practices and may be intentional or unintentional.

Unethical or Illegal actions: Unethical or illegal actions are defined as a gross deviation from GeoLabs standard operating procedure specified analytical practices, combined with the intent to conceal the deviation. The difference between poor analytical judgement and fraud may be assessed in the documentation of intent within
laboratory records. Gross deviations from specified procedures will be investigated for potential fraud, and findings of fraud will be dealt severe penalties.

Examples of Improper/Unethical/Illegal Practices:

- Improper use of manual integrations to meet calibration or method QC criteria (e.g., peak shaving or peak enhancement are considered improper, unethical, or illegal actions if performed solely to meet QC requirements).

- Time travel of analyses to meet method requirements (e.g., intentionally resetting a computer system’s or instrument’s date and/or time to make it appear that a time/date requirement was met).

- Falsification of results to meet method requirements.

- Reporting of results without analyses to support (e.g., dry-labbing).

- Selective exclusion of data to meet QC criteria (i.e., initial calibration points dropped without technical or statistical justification.)

- Misrepresentation of laboratory performance by presenting calibration data or QC limits within data reports which are not linked to the data set reported, or QC control limits presented within any document which are not indicative of historical laboratory performance or used for batch control.

- Notation of matrix interference as basis for exceeding acceptance limits (typically without implementing corrective actions) in interference-free matrices (e.g., Method Blank or Laboratory Control Samples).

- Unwarranted manipulation of computer software (e.g., improper background subtraction to meet ion abundance criteria for GC/MS tuning, chromatographic baseline manipulations).

- Improper alteration of analytical conditions (e.g., modifying EM voltage, changing GC temperature program to shorten analytical run time) from standard analysis to sample analysis.

- Misrepresentation of QC samples (e.g., adding surrogates after sample extraction, omitting sample preparation steps for QC samples, over-spiking or under-spiking).

- Reporting the results from the analysis of one sample for those of another.

- Any misrepresentation of practices or procedures used within our company, to any internal or external auditor, by any employee of GeoLabs.

To avoid any miscommunication the basis for manual integrations on QC data shall be clearly documented within the analytical record. Peer review and/or review by the Laboratory Director shall also be documented. All changes in data shall be clearly marked to show who made the change, date changed, and why changed.

An in-depth data audit will be conducted on 5% of all final reports produced. This data review shall include a check of all data for any data integrity issues. All possible data integrity issues will be clearly noted on a Quality Finding Report and forwarded to the President and Laboratory Director. An evaluation will be made regarding the extent of the infraction. A decision will be made by management of training needs or possible penalties.
A proactive program for the detection of improper, unethical, or illegal actions will be included in our yearly technical review. This will include the audit of laboratory records, calibration data, computer back-up records, and manual integrations.

Instances of fraud in the laboratory will result in the dismissal of the guilty party and prompt notification of any affected client or agency.

STATEMENT OF UNDERSTANDING:
I am aware of GeoLabs’ Ethics Policy. I have read the document and any questions I may have had on the issues involved have been answered to my satisfaction. I agree to abide by this policy.

Signed: ___________________________  Print Name: ___________________________  Date: ___________________________
3.0 DOCUMENTATION, DOCUMENT CONTROL, DATA BACKUP AND PROTECTION

Laboratory documentation, such as the Quality Assurance Plan, Chemical Hygiene Plan, and Standard Operating Procedures are perhaps the most important aspect of proper laboratory practices. These documents are controlled by the Quality Assurance Manager. Revision control is implemented through the following philosophy: minor changes/corrections to existing versions are documented within the software by use of revision tracking. Major changes require a new version number and retirement of the previous version. Previous versions will be marked with the initial and final dates of use. Implementation of an historical library of documents was begun in 1999. Hard copy and electronic versions are archived.

Report formats, including appropriate RDLs and method references, are controlled by the Report Specialist in conjunction with the Quality Assurance Manager. Reports are generated within our Khemia Omega LIMS system.

The record keeping system allows historical reconstruction of all laboratory activities that produced the resultant sample analytical data. The history of the sample is understood through the documentation. All documentation entries must be signed or initialed by responsible staff. The reason for the signature or initials must be clearly indicated in the records, such as “sampled by”, “prepared by”, or “reviewed by”.

The laboratory shall retain on record all original observations, calculations and derived data, calibration records and a copy of the test report for a minimum of ten years.

Documentation Control and Archival Practices:

<table>
<thead>
<tr>
<th>Type of Document</th>
<th>Stored</th>
</tr>
</thead>
<tbody>
<tr>
<td>Final Report</td>
<td>Paper version filed with all associated data (instrument reports, calculations, chain of custody, etc.). All are stored on-site. Electronic files associated with the report are archived and stored in two geographically separated locations on external hard drives.</td>
</tr>
<tr>
<td>Instrument Data Files</td>
<td>Electronic data files are archived nightly and stored in two geographically separated locations on external hard drives.</td>
</tr>
<tr>
<td>Quality Control Data</td>
<td>Paper copies of batch quality control data are kept in each analytical area. Electronic files are archived as mentioned above.</td>
</tr>
<tr>
<td>Log books</td>
<td>Log books are issued by the QA manager and are archived by the chemist. Copies of appropriate pages are included with reports.</td>
</tr>
<tr>
<td>IDCs, MDL Studies</td>
<td>Kept on file in QA office method specific files.</td>
</tr>
<tr>
<td>Control Charts</td>
<td>Control Charts can be viewed from within our LIMs system based on entered QC data.</td>
</tr>
</tbody>
</table>

Backup Procedures

All important data files from networked computers are archived daily to a server. The servers are archived on a quarterly basis to an external hard drive, which is kept off-site.
4.0 REVIEW OF ALL REQUESTS, TENDERS AND CONTRACTS

All new work is initiated by the Laboratory Director who delegates responsibilities for the new work according to available resources. Affected staff members meet prior to initiation of new work in order to determine if appropriate facilities and resources are available. The plan for any new testing shall be reviewed and approved by the Laboratory Director before commencing such work. If the review uncovers any potential conflicts, deficiencies, inappropriate accreditation status, and/or inability to perform the work, the laboratory shall notify the client. In cases where differences exist between the request/tender and contract they shall be resolved prior to starting work.

The review shall document that facilities and resources are organized to efficiently perform the work, including subcontracted work. The record of contract review includes pertinent discussions with the client regarding their requirements and results submitted during the contract period. For routine reviews of ongoing work a date and a signature of the laboratory official responsible for the contract is sufficient. For any new testing requirements, the designated official shall ensure that standard operating procedures and demonstration of capability to perform those tests prior to reporting results are available. The SOP’s shall be under document control and a Demonstration of Capability statements shall be on file.

Clients are notified immediately in situations where the laboratory cannot conform to the contract and if there is a change in laboratory accreditation status.

5.0 SAMPLE RECEIPT, CUSTODY AND LABORATORY OPERATIONS

5.1 Sample Containers

GeoLabs supplies new bottles for sample collection. GeoLabs customers are supplied with appropriate sample containers in order to maintain the integrity of each sample in accordance with EPA sampling protocols. Requests for sample bottles are generally made by telephone from the prospective client and are assembled by the sample custodian. Containers are supplied with labels, preserved if appropriate, and packed in coolers for delivery by GeoLabs’ courier. A Chain of Custody form is supplied to the customer with the bottle shipment. The containers are then shipped by the appropriate method to the client. Approximately 95% of the sample bottles are hand delivered to the client by the GeoLabs’ courier. Trip blanks (containers filled with laboratory DI water) are shipped or hand-delivered with sampling bottles at random to monitor “trip” conditions. Customers may also request trip blanks.

Metals analysis grade 1:1 nitric acid, 1:1 sulfuric acid and 1:1 hydrochloric acid are supplied by the laboratory. Clients can request to have in-house preserved bottles dropped off by the laboratory courier. The sample custodian preserves the requested bottles in the laboratory before delivery.

Records of container shipments, purchase orders, shipping documents, and related paperwork are maintained by office personnel. Shipping documents may include manufacturer’s lot numbers, certification statements, point of origin, shipping dates and specific quantities. The date of physical receipt of all bottle shipments are written in indelible ink directly on the shipping invoice. The shipping invoice is also initialed by the individual who accepted the delivery.

Sample containers, preservation and holding times are documented in Appendix C.

5.2 Chain of Custody
All samples received by the Laboratory must be accompanied by a properly filled out Chain of Custody. An example of GeoLabs' Chain of Custody is given at the end of this section. The Chain of Custody form identifies the client's samples, special conditions, and the tests to be performed. Chain of custody forms contain all of the information referred to by "Test Methods for Evaluating Solid Wastes; Physical and Chemical Methods", SW-846, (U.S. EPA, 1982b). Specifically:

- Client’s name, address, phone and fax numbers.
- Person to contact with questions.
- Name of project and billing information
- Expected turn-around time
- Special instructions
- Client sample identification
- Date and time of sampling, initials of person taking sample, if more than one
- Type of sample
- Type of container
- Number of containers
- Preservative
- Tests desired
- Space for lab pH and temperature of sample when received
- Signature of client or client's agent
- Signatures of all intermediate recipients of the samples

All incoming samples are checked to verify sample integrity, i.e. samples are on ice, no headspace for VOAs, have been preserved properly, and complete documentation and identification are present. A temperature check, by use of a non-contact infrared thermometer, is performed on all samples at time of receipt, and recorded on the chain of custody. In addition, the tests requested are reviewed and the submitted sample volumes, container types and preservation are verified to make sure that they are appropriate for the testing requested. Should there be any question concerning whether the laboratory can perform the requested test, the issue is addressed with the Laboratory Director or the QA Manager before the samples are accepted.

5.3 Sample Receipt and Storage

Chains of Custody are inspected by login personnel to ensure that the chains are correctly filled out and that the tests requested are appropriate. A temperature check is performed on all samples and included on the chain of custody. The pH of samples is determined by transferring a small amount of sample by disposable pipette onto a wide range pH paper and the result is included on the chain of custody (excluding volatile, soil, and Oil and Grease samples).

Login personnel, office personnel cross-trained for login, the Lab Director, QA Manager, and President may all accept samples.

Unique sequential sample numbers are given to each sample. These sample numbers are recorded in ink directly on the original Chain of Custody. These numbers are written on all sample containers (both the jar and the cap) in indelible ink. All samples received at GeoLabs are logged into the LIMS. This data base, along with the Chain of Custody, serves as the reference for each sample in terms of the sample number, client's name, sample type, analyses requested, date received, date collected, and appropriate comments. In addition, as required by the Massachusetts DEP, a hand-written log of samples received, lab identification numbers assigned and test requested is maintained. Each entry is initialed by the person entering the data. After a set of samples has been numbered and entered into the computer, copies of the Chain of Custody are distributed to each analytical department that will be involved in the analysis, and to the accounting department. A project folder is prepared with client name, sample numbers and internal due date. The Chain of Custody remains in this folder until project completion.
Samples are stored in accordance with the guidelines in Appendix C and Section 4.9 of this manual. Several refrigerators and freezer units are used to segregate samples from analytical standards. Analytical standards for volatile, semi-volatile and pesticides/PCB analyses are stored separately in three refrigerator/freezer units. Samples and extracts are kept in refrigerator units. These refrigerators are monitored daily to insure a constant temperature of 4° ± 2° C. Provision has been made for isolated storage for volatile organic samples. All volatile organic samples, including duplicates, field blanks, and trip blanks, are stored together in the same refrigerator and maintained at 4.0° ± 2° C. All other samples are excluded from the volatile organic refrigerator. Upon receipt, samples are placed in a secure storage area to which only the Laboratory Personnel have access. After an analyst has removed an appropriate aliquot of the sample, any remaining sample is returned to the storage area. The analyst is responsible for maintaining proper custody of the sample aliquot and extracts.

5.4 Holding Time Confirmation

Holding time confirmation is verified from the Chain of Custody supplied with the sample. The Sample Custodian reviews the chain and makes note of the sampling date, sampling time and the date samples were received by the laboratory. The holding time is then calculated from the actual time of sample collection and correlated with the recommended EPA holding time specified in Appendix C of this manual. Samples that are rejected due to holding time violations are documented by writing “Holding Time Exceeded” in ink in the comments section of the chain of custody sheet. The client is contacted and a new sample is requested. In some cases where there is limited sample, the client may request that the analysis be performed despite the holding time issue. These exceptions are noted on the Chain of Custody and in the Final Report.

5.4.1 Protocol For Sample Rejection

The following deficiencies are grounds for sample rejection:
1. Incomplete or poorly filled out chain of custody form.
2. Lack of collection dates and collection times
3. Sample supplied without chain of custody form(s)
4. Lack of authorized signatures
5. Samples collected and stored by client that are beyond EPA holding times
6. Samples that are collected and stored in vessels that are not within EPA specifications. Examples: VOA samples received in plastic containers or TPH samples contained in propylene bottles.
7. Broken or damaged sample containers resulting in the loss of sample integrity.
8. Samples that contain insufficient sample volume or weight for a given analysis.

In all cases, where there is a perceived problem with a sample or with the chain, the client is notified and the problem and potential solutions are discussed. At the client’s request, GeoLabs will proceed with an analysis, but will note the problem in the Final Report.

5.5 Laboratory Documentation

Many documents facilitate the traceability of data, if necessary, through each step of sample handling to provide valid evidence should such data become the subject of testimony. These documents include the laboratory logbooks, laboratory data records, correspondence, reports, sample tags, chain of custody records, bench sheets, and instrument reports. Entries into these documents are initialed by the responsible individuals and reviewed, where appropriate. These documents (or copies of the appropriate pages) are kept with the Final Report for each project.

Laboratory documentation involves three general categories:
- Sample flow
- Analytical activities (e.g. instrument calibration, reagents etc.)
• Data reduction
Sample flow documentation is summarized below and the remaining two items will be discussed in detail in
subsequent sections.

All references to a sample are made using the GeoLabs sample identification number, and all beakers, containers,
etc. are marked similarly, unless clearly impossible. In the latter case, apparatus numbers are cross-referenced with
sample numbers in the analyst's notebook. The following list describes the records associated with sample handling
at GeoLabs:

1. The Chain of Custody documents the history of the sample. The Chain of Custody form also provides the
analysts with the necessary information to complete each test.
2. The LIMS is the basic reference for information on all samples received by GeoLabs.
3. Sample numbers affixed to each container are unique to that sample and continuously increment as samples are
received.
4. A daily Job Track is maintained in the LIMS to keep a running log of all work in progress. Information is
organized according to client, collection date, analysis due date, report due date, sample numbers, and
appropriate comments.
5. As work on the sample progresses, instrument reports and related raw data are maintained in a project folder.
6. A new chain of custody is initiated when a sample is sent to a subcontract laboratory for analysis. GeoLabs'
  sample number is used as the sample identifier and GeoLabs is listed as the client in order to maintain the
  confidentiality of our client. This chain of custody shall also indicate the state regulatory criteria that must be
  followed by the subcontracted laboratory. When samples are sent to local subcontract labs for analysis,
  GeoLabs’ courier delivers the samples and maintains the custody record. When samples are shipped, they are
  sent via Fed-ex, carefully packaged, and the shipping number is used for sample tracking.

5.6 Forms

Master copies of forms are maintained in an active computer data base and hard copies are stored in separate files
that are accessible by the laboratory staff. When additional laboratory forms are required, a hard copy is obtained
from the filing system and reproduced.

5.7 Storage of Hard Copy

Hard copies of laboratory reports are filed and indexed sequentially by their LIMS generated identification number
and are stored in file cabinets. Each laboratory report is accompanied by all bench sheets, calculations,
quantification reports, chromatograms, correspondence relating to laboratory activities for the project, and chain of
custody sheets. Hard copies are maintained on-site for ten years after which they will be disposed of. All
documents will be retained for a minimum of ten years.

5.8 Final Reports

Hard copies are generated by the Reports Specialist using our LIMS system. When all analyses are complete, the
project folder is assembled by the Sample Custodian and given to the Reports Specialist. The Reports Specialist
verifies that the appropriate testing has been performed and provides a review of the data for completeness. The
appropriate parameters are checked against the analysis raw data to verify correct entry into the LIMS by the
analyses. The report is then printed, proofread and submitted to the Quality Assurance Officer for initial review.
Calculations are checked for accuracy and detection limits are confirmed. All data points on the completed reports
are compared with the data points on the bench sheets. The report is then submitted to the Laboratory Director for
final review. Approval is documented with the Laboratory Directors signature and forwarded to the Report
Specialist for distribution. The final report and a copy of the chain of custody form are e-mailed to the customer. Final reports may also be faxed or e-mailed at the client’s request.

Reports for all Massachusetts DEP reportable work for potable and non-potable water will include a copy of our Massachusetts DEP certified parameter list.

All samples submitted for reporting under a program that requires that a certified laboratory perform such testing, and for which Geolabs does not hold current certification, will be sub-contracted to a certified laboratory. All reported analysis which is sub-contracted will be clearly labeled with the sub-contractors laboratory ID number.

5.9 Analysis Scheduling

Some samples and analyses are sensitive to sample storage time. Priority is given to time-sensitive samples. Such priority is initiated and tracked through the LIMS functions. More routine analyses are performed according to the individual analyst’s overall job schedule, always with the report due date in mind. The flow of samples which require several steps of processing prior to final analysis is scheduled by the Extractions Supervisor.

5.10 Sample Storage and Disposal

Prior to analysis, samples are stored so as to preclude alteration in any way from their condition as received. After receipt, samples are stored for 30 days, in case analyses must be rerun. The chemical integrity of the samples is maintained by the addition of appropriate preservatives at the time of collection and maintaining appropriate physical conditions such as low temperature.
**GeoLabs, Inc.**

**Environmental Laboratories**
45 Johnson Lane
Braintree, MA 02184
Office: 781-848-7844 Fax: 781-848-7811

**Turnaround Time**

<table>
<thead>
<tr>
<th>RUSH:</th>
<th>24hrs</th>
<th>48hrs</th>
<th>72hrs</th>
<th>STANDARD:</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>5 Days</td>
<td>Rush</td>
<td>Approved</td>
<td>By: _____</td>
</tr>
</tbody>
</table>

**SPECIAL INSTRUCTIONS**

**Client:**

**Address:**

**Phone:**

**Fax:**

**Contact:**

**Project Number:**

**Project Location:**

**Purchase Order #:**

**Collected By:**

**ANALYSES REQUESTED**

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>COLLECTION</th>
<th>SAMPLE LOCATION</th>
<th>CONTAINER</th>
<th>TYPE</th>
<th>QUANT</th>
<th>MATRIX</th>
<th>COMP</th>
<th>GRAB</th>
<th>PRES</th>
<th>GeoLabs SAMPLE NUMBER</th>
</tr>
</thead>
</table>

<table>
<thead>
<tr>
<th>SAMPLE CODES:</th>
</tr>
</thead>
<tbody>
<tr>
<td>A = Amber</td>
</tr>
<tr>
<td>B = Bag</td>
</tr>
<tr>
<td>G = Glass</td>
</tr>
<tr>
<td>P = Plastic</td>
</tr>
<tr>
<td>S = Summa Canister</td>
</tr>
<tr>
<td>O = Other</td>
</tr>
<tr>
<td>V = VOA</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>MATRIX CODES:</th>
</tr>
</thead>
<tbody>
<tr>
<td>GW = Ground Water</td>
</tr>
<tr>
<td>WW = Wastewater</td>
</tr>
<tr>
<td>DW = Drinking Water</td>
</tr>
<tr>
<td>SL = Sludge</td>
</tr>
<tr>
<td>S = Soil</td>
</tr>
<tr>
<td>A = Air</td>
</tr>
<tr>
<td>O = Oil</td>
</tr>
<tr>
<td>OT = Other</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>PRESERVATIVE CODES:</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 = HCl</td>
</tr>
<tr>
<td>2 = HNO₃</td>
</tr>
<tr>
<td>3 = H₂SO₄</td>
</tr>
<tr>
<td>4 = Na₂S₂O₃</td>
</tr>
<tr>
<td>5 = NaOH</td>
</tr>
<tr>
<td>6 = MeOH</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Relinquished By:</th>
<th>Date/Time</th>
<th>Received By GeoLabs:</th>
<th>Date/Time</th>
</tr>
</thead>
</table>

**GEOLARS CHAIN OF CUSTODY**
6.0 Analytical Procedures

Many procedures performed at GeoLabs are highly specific to a particular type of analysis or instrument. All such procedures are maintained on file in our laboratories and are accessible to all chemists. All analyses are performed in accordance with EPA, NIOSH, MA DEP, Standard Methods or ASTM protocols. Standard Operating Procedures (SOPs) for each analysis are maintained on file. The SOPs contain all instructions, references, deviations from methods, and contingency measures. SOPs are updated whenever changes in the methodology are made.

6.1 Glassware

Inorganic/Metals Laboratory
Glassware is washed with detergent (Liquinox for inorganics, Citranox for metals), rinsed with hot tap water and then rinsed three times with distilled, deionized water (15 Mohm resistivity or higher) (DI). Glassware used in trace metals analysis is also rinsed with a 1:1 nitric acid-water mixture followed by a thorough rinsing with distilled, deionized water. Separate glassware is reserved for use in the analysis of phosphorous. Glassware is then covered and stored in the area where it will be used.

Organic Laboratory
Glassware is washed with Liquinox detergent, rinsed with hot tap water and then distilled water. The glassware is then allowed to air dry and is stored in laboratory drawers and cabinets. Certain glassware is baked out in muffle furnaces or drying ovens. Volumetric glassware is never baked. When ready for use, the chemist rinses the glassware with the appropriate solvent for the test being performed.

6.2 Reagent Preparation and Storage

Solvents used in the preparation of reagents and standards include distilled, deionized water and high purity organic solvents (glass-distilled, pesticide grade or re-distilled, when necessary). When new shipments of reagents are received by the laboratory, all shipping invoices and associated papers are placed into files which are arranged alphabetically by manufacturer. Lot numbers are recorded in the Chemical Receipt book. When reagents and solvents are received they are labeled with the receipt date. When they are opened, they are labeled with the “date opened”. These notations are made on the product label using indelible ink.

All reagent solutions used are prepared from Analytical Reagent Grade (AR) chemicals or higher purity grades, as required by the stated method. The preparation of all reagent solutions is documented in the appropriate laboratory notebook, including source, weights and dilutions. Each reagent solution is clearly labeled with the composition, concentration, date prepared, initials of preparer, expiration date, and special storage requirements if any.

Reagent solutions are stored in appropriate glass, plastic or metal containers. Reagents are stored under conditions designed to maintain their integrity (refrigerated, dark, etc.). Shelf life is listed on the label and the reagent is discarded after it has expired. Dry reagents such as sodium sulfate and silica gel are baked at 400°C and rinsed with solvent prior to use for organic chemical analyses. See Table 5.1, Reagent and Standard Storage.

6.3 Training

At GeoLabs, great effort is made to maintain a staff of scientists of exceptional quality. The Laboratory Director certifies that personnel with appropriate educational and/or technical background perform all tests for which the laboratory is accredited. During the interview process, the Laboratory Director reviews each prospective employee's educational experience, basic technical background, and/or prior professional experience, to insure that they are capable of understanding and following the methodology that will be their responsibility. Upon hiring a new analyst, and upon change of positions of existing employees, GeoLabs will proceed with a training program to acquaint that individual with any unfamiliar analytical procedures, in-house
practices and our QA/QC requirements. Employees are provided a certification statement that ensures that they have read, understand and agree to follow this QA Plan, the latest version of the Standard Operating Procedures that relate to their job function, and the laboratory Chemical Hygiene Plan. Employees will also be provided with our Ethics Policy, which will describe their ethical and legal responsibilities.

The performance of the individual is evaluated after a 90-day trial period. Each new employee is expected to be completely familiar with the QA Plan, Chemical Hygiene Plan, and applicable SOPs by the end of that period. In addition to their supervisor, the Laboratory Director and QA Manager are available to the new analyst at any time to discuss all of GeoLabs in-house procedures and requirements. For all technical staff involved with the testing of environmental samples a Demonstration of Capability is required, the results of which will be kept on file.

In addition to this initial training, all employees of GeoLabs are urged to attend short courses available from instrument manufacturers, professional groups, seminars and other methods of improving employee performance and knowledge.

Continued proficiency will be documented on a yearly basis by checking analysts results on four consecutive laboratory control samples with acceptable levels of precision and accuracy. If this is not possible, documentation of acceptable performance of a blind sample will be used.

All personnel training records will be kept in a secure centralized file, and maintained by the Quality Assurance Manager. These records will include any documentation of training courses or workshops on specific equipment, analytical techniques or laboratory procedures. This file will also contain all certification statements signed by personnel.

6.4 Safety
The safety of its employees and neighbors is a prime concern of GeoLabs. Safe practices not only ensure individual and environmental protection but also result in better quality work. Hence GeoLabs has a Chemical Hygiene Plan and Emergency Plan which cover the following topics:

- Housekeeping
- Labelling/Signs
- Protective Equipment/Apparel/Personal Habits
- Accident Reporting
- Injuries
- Fires
- Refrigerators/Freezers
- Ventilation
- Emergency Procedures
- Waste Disposal and spill cleanup

6.5 Waste Disposal
Samples are packaged, categorized and disposed of according to the applicable EPA and MA DEP Hazard Class. GeoLabs is considered a small quantity generator, therefore, drinking water samples and non-hazardous wastewater, groundwater and surface water samples with a pH less than 5 or greater than 9 are disposed of by neutralizing to a pH of 5-9 and discharging under our industrial permit. Contaminated soil samples are bulk stored in 17H drums in GeoLabs' hazardous waste storage area for disposal by an approved disposal company. Sample extracts are disposed of in the appropriate waste barrels (ie. acid/metals waste, organic solvent waste, cyanide waste, mercury waste, and non-RCRA, non-hazardous soil waste).
Laboratory Waste is separated into seven groups:

1. Acid/Metals Waste: generated during the preparation and analysis of samples and standards for metals analyses, as well as the disposal of client samples that contain metals concentrations over regulated levels.
2. Cyanide Waste: generated during the analysis of samples and the preparation of standards for cyanide analysis, the use of cyanide containing reagents for other analyses, and the disposal of cyanide contaminated samples.
3. Waste Oil / Organic Solvent Waste: generated from the analysis of waste oil samples and during the analysis of PCB's, pesticides, volatile and semi-volatile organics, MBAS, TPH, the disposal of organic extracts, and the solvents used for cleaning glassware. (PCBs and pesticides below regulated levels)
4. Soil Waste: generated during the analysis of soil samples. GeoLabs disposes of these samples as a service to our clients to ensure that they are disposed of properly.
5. Stannous Chloride Waste: Generated during the analysis of metals. GeoLabs disposes of stannous chloride waste in a drum located in the hazardous waste storage room.
6. Pyridine Barbituric Acid Waste: Generated by Wet Chemistry during the analysis of reactive cyanides, total cyanides, etc. GeoLabs disposes of pyridine-barbituric acid waste into the solvent waste drum located in the hazardous waste storage room at a rate of 2 gallon per 53 gallons solvent waste.
7. Poly-Chlorinated-Biphenyl (PCB) Waste: Generated during the extraction of PCBs. Disposed of into a waste drum located in the hazardous waste storage room.

Waste is stored in a special locked waste storage room accessible from outside of building only. Keys to this room are monitored. The waste room and its contents are marked and labeled according to EPA Hazardous Waste Activity Protocols. Waste generated during the course of analyses is initially stored in properly-labeled gallon waste disposal bottles, which are placed in marked cabinets in the laboratory satellite waste areas. Proper labeling includes the use of a hazardous waste sticker with the full chemical name, the start date of the bottle, the EPA waste code number, the in-house number, and the company name. The in-house number is obtained from the “generated waste” log book, in which the labeling information is also entered. Waste is emptied a minimum of once per week or more often if needed into the appropriate waste barrel in the storage room and logged into the generated waste log book. This procedure is performed by an OSHA-trained chemist wearing proper safety clothing. The chemist is always accompanied by a safety observer.

Waste is disposed of by a licensed hauler and taken to licensed waste disposal facilities. These procedures are tracked on Uniform Hazardous Waste Manifest Sheets (EPA Form 8700-22, Rev. 9/94).  

**TABLE 5.1**  
**REAGENT AND STANDARD STORAGE**

<table>
<thead>
<tr>
<th>Chemical</th>
<th>Method of Storage</th>
</tr>
</thead>
<tbody>
<tr>
<td>Acids (Nitric, Sulfuric, Hydrochloric, Acetic)</td>
<td>Stored in original containers in vented cabinets designed for acid storage.</td>
</tr>
<tr>
<td>Organic Solvents</td>
<td>Stored in original containers in vented solvent storage cabinets. All solvents used for VOC analyses are stored separately in the VOC analysis area.</td>
</tr>
<tr>
<td>Inorganic Chemicals, Reagents and Standards</td>
<td>Stored in cabinet designated for standard and reagent storage. Cabinet is in temperature controlled area of the laboratory. Certain heat and/or light sensitive reagents are stored according to manufacturer’s instructions.</td>
</tr>
<tr>
<td>pH Buffers</td>
<td>Stored in designated reagent storage cabinet in a temperature controlled area of lab.</td>
</tr>
<tr>
<td>Organic Standards</td>
<td>Stored in designated Refrigerator/freezer, i.e. VOC standards are stored separately from Semi-VOA and PCB standards.</td>
</tr>
</tbody>
</table>
6.6. Chemical Handling Procedures

The following instructions are given to all chemists and others who come in contact with chemicals:

All chemicals used in the laboratory should be handled with care at all times. Gloves, safety glasses and lab coats should be worn at all times while in the laboratory. The following guidelines should be followed:

Any solvent use should take place in the hoods. Solvent vapors are very harmful and should not be inhaled under any circumstances. Carry bottles with two hands. Use care when pouring into squirt bottles. Avoid skin contact. Change gloves immediately if contact occurs. Clean up spills with towels or absorbent. Allow empty bottles to vent before throwing away.

All acid bottles should be carried with two hands, and dispensed or poured in a hood. Always add acid to water, and never the reverse. Use caution to avoid spilling or splattering. If acid gets on gloves or lab coat, change them immediately. Clean up small spills with absorbent or neutralizer. Rinse empty bottles into the acidic rinse water waste before throwing away.

All other hazardous reagents and chemicals should be handled with caution at all times. If you are ever unsure about the hazards of any chemical, obtain the MSDS and read it thoroughly. All MSDS sheets are located in the QA Managers office. If you are still in doubt, ask someone for help to be sure you are handling the chemical properly. Again, ALWAYS wear gloves, safety goggles, and a lab coat. More detailed instructions are given in the Chemical Hygiene Plan.
7.0 CALIBRATION

Calibrations substantiate the precision and accuracy of routine sample analysis. All instruments and equipment must be calibrated and pass the calibration standards set by method quality control before samples can be run with any merit. Calibration criteria can be found in Table 7.3. Reference thermometers and reference weights must be re-calibrated every 5 years as required by MADEP regulations.

7.1 Standard Receipt and Traceability

Standards are received by the chemist in charge of the analysis to be performed and logged into the New Standard Log Book. In the New Standard Log Book, the standard's compounds, the date received, and lot number are entered. The Certificate of Analysis is then entered into the appropriate notebook.

Organics Laboratory

Mixed or individual concentrates and neat standards are purchased from certified suppliers in sealed ampoules or vials. Each standard comes with a Lot Analysis Sheet and lot number. The standard is dated and stored as recommended by the manufacturer. Certification and traceability statements are kept in a binder in the appropriate laboratory.

Metals Laboratory

Individual standards are purchased from the supplier. Lot number and expiration dates are on each label. The standard is received by the metals chemist, dated and stored according to the manufacturer's instructions. Records of Certification and Traceability are on the label and therefore kept with the stock standard.

Inorganics Laboratory

Standards are purchased from certified commercial suppliers. Standards and reagents are received by the Inorganic Laboratory chemist. They are dated and stored according to the manufacturers instructions. Only high quality, certified chemicals are purchased. Certification, Lot Analysis and Traceability are on the label.

7.2 Standards Sources, Preparation and Storage

Quantitative reagents are prepared from neat materials or concentrates supplied by certified suppliers, and are standardized or checked against primary standards, check standards, or by other appropriate techniques to ensure the reliability of the reagent. They are prepared using analytical balances, Class A volumetric glassware, with utmost care and proper technique, and are stored in appropriate containers under specific conditions.

Working standards are prepared from stock solutions as needed. Working standards can be traced to stock standards by lot number, date and analyst initials. Preparation of working standards is recorded in the analyst's laboratory notebook. Lot number, date of expiration, date of preparation, weight, volumes, chemist name, lab ID number, and solvent type are all recorded in the notebook. The standard container is labeled with the contents, lot number, date of preparation, expiration date, and analysts initials. See Table 6.2 Standard Sources and Preparation.

7.3 Instrument Calibration

See Table 6.3 Instrument Calibration

7.4 Method Calibration Requirements

Method calibration requirements detailed in this manual are based on methodologies established by the U.S. EPA and the State of Massachusetts Department of Environmental Protection.
Given that more stringent calibration methodologies may be required for a given client, GeoLabs will implement, adhere, and follow these new calibration procedures in their entirety. Modifications will be incorporated into all forms and documents utilized in the method calibration process. These modified documents and the modified procedural formats will be recorded, filed, and maintained within the existing method calibration filing system.

7.5 Documentation and Records

Documentation and records of calibration are contained and stored within laboratory notebooks which are maintained by each analyst. Instrument calibrations are conducted before each analysis series within the wet chemistry and metals analysis divisions. Calibrations for the GC/MS instruments are instituted every 12 hours. The GC-FID and GC-ECD units are calibrated as specified by the particular method. A hard copy of each calibration is maintained by the chemist responsible for each instrument.

7.6 Calibration Procedures for GC/Mass Spec

BFB/DDTPP tuning is initiated every twelve hours. Tuning criteria are based on ion abundance criteria, % relative abundance, % relative standard deviation of response factor, base peak and appropriate peak. Acceptance and rejection ranges for the tuning procedure are detailed in the Ion Abundance section of the BFB/DDTPP performance standards forms. If the instrument fails to pass the stated tuning parameters, then a fail tune notation will appear in the status column of the BFB/DDTPP performance standard forms. After tuning, the check calibration standard is injected every 12 hours. A fail tune notation requires the analyst to examine the GC section, MSD section and additional components to determine the cause of failure. Once defined, the cause of the tuning failure is corrected, a notation is then made in the GC/MS operator’s laboratory notebook. The operator notes the corrective actions that were required to remedy the tuning failure in the maintenance notebook. The tuning procedure continues to be conducted until tuned status is achieved.

7.7 Method Detection Limit (MDL)/ Limit of Detection (LOD)

The terms Method Detection Limit (MDL) and Limit of Detection (LOD) are interchangeable. LOD’s are initially determined in accordance with 40 CFR Part 136, Appendix B. Massachusetts DEP requires that LOD’s be prepared and analyzed over a 3 day period, and utilizing all instrumentation used for each specific method. Updating of LOD (MDL) data will be conducted once yearly for all certified analytes or whenever a major change occurs. An LOD will not be necessary for methods where no spiking solutions are available, such as pH. The LOD results must be lower than the Limit of Quantitation (LOQ). Where a multi-level calibration is utilized the LOQ is set at the low standard of the calibration. On other analyses the LOQ will be a value between 2 to 5 times the LOD. Once a LOD is established, it must be verified, on each instrument, by analyzing a quality control sample (prepared as a sample) at approximately 2-3 times the calculated LOD for single analyte methods, and 1-4 times the calculated LOD for multiple analyte methods. The LOD is verified when the QC sample is qualitatively identified. When an LOQ is established, it must be initially verified by the analysis of a low level QC sample at 1-2 times the LOQ, and annually thereafter. A verified LOQ will be one where the recovery of the QC sample is +/- 50%.
## Instrumentation List

### Laboratory Equipment

<table>
<thead>
<tr>
<th>Equipment</th>
<th>Number</th>
<th>Make and Model</th>
<th>Approx. age (yrs)</th>
</tr>
</thead>
<tbody>
<tr>
<td>GC-ECD (2 ECD's)</td>
<td>2</td>
<td>Hewlett Packard 6890</td>
<td>9</td>
</tr>
<tr>
<td>GC-FID</td>
<td>2/2</td>
<td>Hewlett Packard 6890/5890</td>
<td>9</td>
</tr>
<tr>
<td>GC-FID/PID</td>
<td>1</td>
<td>Hewlett Packard 5890</td>
<td>2-6</td>
</tr>
<tr>
<td>GC/MS</td>
<td>4</td>
<td>Hewlett Packard 6890/5973</td>
<td>4-5</td>
</tr>
<tr>
<td>Ultrasonic Cleaner</td>
<td>3</td>
<td>Americanbrand/VWR/Fisher</td>
<td>7</td>
</tr>
<tr>
<td>ICP</td>
<td>2</td>
<td>Perkin Elmer P400/Leeman</td>
<td>2-7</td>
</tr>
<tr>
<td>Graphite Furnace/AA</td>
<td>1</td>
<td>Perkin Elmer Aanalyst 800</td>
<td>3</td>
</tr>
<tr>
<td>GC/MS</td>
<td>2</td>
<td>Hewlett Packard 5972/6890 Series</td>
<td>2</td>
</tr>
<tr>
<td>Air Concentrator with Autosampler</td>
<td>1</td>
<td>Entech</td>
<td>2</td>
</tr>
<tr>
<td>Air Canister Cleaning System</td>
<td>1</td>
<td>Entech</td>
<td>2</td>
</tr>
<tr>
<td>Air Standard Diluter</td>
<td>1</td>
<td>Entech</td>
<td>2</td>
</tr>
<tr>
<td>Tekmar Precept II Autosampler</td>
<td>1</td>
<td>Tekmar</td>
<td>2</td>
</tr>
<tr>
<td>CEM Mars X Microwave Extractor</td>
<td>1</td>
<td>CEM</td>
<td>2</td>
</tr>
<tr>
<td>Lachat Quik-Chem Flow Injector Analyzer</td>
<td>1</td>
<td>Zellweiger Analytical</td>
<td>2</td>
</tr>
<tr>
<td>Analytical Balance</td>
<td>3</td>
<td>DenverInst.Co./Mettler/AE160/AE200</td>
<td>3-6</td>
</tr>
<tr>
<td>pH Meter</td>
<td>2</td>
<td>Accumet Model 15/Orion EA-920</td>
<td>2-6</td>
</tr>
<tr>
<td>Circulating Hot Air Oven</td>
<td>1</td>
<td>Labline 3605-M</td>
<td>6</td>
</tr>
<tr>
<td>Muffle Furnace</td>
<td>3</td>
<td>Thermolyne 1400/Lindberg/51848</td>
<td>6-11</td>
</tr>
<tr>
<td>Conductivity Meter</td>
<td>1</td>
<td>Cole Parmer 1500-20</td>
<td>8</td>
</tr>
<tr>
<td>Hot Water Bath</td>
<td>2</td>
<td>Precision Electric 9818-R30</td>
<td>11</td>
</tr>
<tr>
<td>Flash Point Tester</td>
<td>2</td>
<td>Koeler K-14600 /Pensky Martin</td>
<td>6</td>
</tr>
<tr>
<td>Vacuum Pump</td>
<td>2</td>
<td>Sklar 100-15/Gast G582GDX</td>
<td>7 &amp; 2</td>
</tr>
<tr>
<td>High Speed Centrifuge</td>
<td>1</td>
<td>Damon/IEC GN-S</td>
<td>7</td>
</tr>
<tr>
<td>Drying Oven</td>
<td>2</td>
<td>VWR 1360/1305U</td>
<td>8</td>
</tr>
<tr>
<td>Visible Spectrophotometer</td>
<td>2</td>
<td>Gensys 20 / Hach DR2000</td>
<td>1 / 6</td>
</tr>
<tr>
<td>Top Load Balance</td>
<td>4</td>
<td>Ohaus</td>
<td>3-6</td>
</tr>
<tr>
<td>Turbopav II Concentrator</td>
<td>3</td>
<td>Zymark</td>
<td>2</td>
</tr>
<tr>
<td>Liquid/Liquid 3D Shaker</td>
<td>1</td>
<td>Glas-Col</td>
<td>3</td>
</tr>
<tr>
<td>Labline 6 Station Hotplate</td>
<td>3</td>
<td>Labline</td>
<td>6</td>
</tr>
<tr>
<td>100 Station Autosampler</td>
<td>1</td>
<td>Centurion from EST</td>
<td>1</td>
</tr>
<tr>
<td>51 Station Autosampler</td>
<td>2</td>
<td>Archon from Varian</td>
<td>11</td>
</tr>
<tr>
<td>Sample Concentrator</td>
<td>4</td>
<td>Tekmar 3000</td>
<td>6</td>
</tr>
<tr>
<td>Orbit Shaker</td>
<td>1</td>
<td>Labline</td>
<td>6</td>
</tr>
<tr>
<td>Chiller</td>
<td>2</td>
<td>Neslab CFT-25/75</td>
<td>2-8</td>
</tr>
<tr>
<td>Flow thru Cooler</td>
<td>1</td>
<td>Neslab EN-150</td>
<td>6</td>
</tr>
</tbody>
</table>
### TABLE 7.2 STANDARD SOURCES AND PREPARATION

<table>
<thead>
<tr>
<th>Instrument</th>
<th>How Received</th>
<th>Source/ Storage</th>
<th>Preparation from Source</th>
<th>Lab Stock Storage</th>
<th>Preparation Frequency</th>
</tr>
</thead>
<tbody>
<tr>
<td>GC (PCB/Pest)</td>
<td>Neat or 2000 µg/mL</td>
<td>Frozen/ Frozen</td>
<td>Primary Standard prepared from Source Stock</td>
<td>4°C</td>
<td>Semi-Annual or when degradation is apparent</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Working Standard from primary</td>
<td></td>
<td></td>
</tr>
<tr>
<td>GC/MS (VOA)</td>
<td>Neat or 2000 µg/mL</td>
<td>Frozen/ Frozen</td>
<td>Primary Standard from Source</td>
<td>4°C</td>
<td>Monthly or when degradation is apparent</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Working Standard from Primary</td>
<td></td>
<td>Weekly</td>
</tr>
<tr>
<td>GC/MS (Semi-VOA)</td>
<td>Neat or 2000 µg/mL</td>
<td>Frozen/ Frozen</td>
<td>Primary Standard prepared from Source Stock</td>
<td>4°C</td>
<td>Monthly</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Working Standard from Primary</td>
<td></td>
<td></td>
</tr>
<tr>
<td>ICP</td>
<td>1000 mg/L</td>
<td>RT</td>
<td>Primary Standard from Source (20 ppm, then 1-10 ppm range)</td>
<td>0.15% HNO₃ 4°C</td>
<td>Annually or as needed</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Working Standard from Primary</td>
<td></td>
<td></td>
</tr>
<tr>
<td>pH Meter</td>
<td>4, 7, 10 Buffer solns</td>
<td>RT</td>
<td>As Received</td>
<td>N/A</td>
<td>Replaced Daily</td>
</tr>
<tr>
<td>Visible Spectrometer</td>
<td>Neat</td>
<td>As directed</td>
<td>Stock Standard from Neat</td>
<td>Refrig 4°C</td>
<td>Monthly or as directed by method</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Intermediate Standard from Stock</td>
<td></td>
<td>Weekly or as directed</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Working Standard from Intermediate Stock</td>
<td>Refrig 4°C</td>
<td></td>
</tr>
<tr>
<td>IR Spectrometer</td>
<td>Neat</td>
<td>Freezer</td>
<td>Stock from Neat Compound</td>
<td>Refrig 4°C</td>
<td>Semi-Annually</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Intermediate Standard from Stock</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Working Standard from Intermediate stock</td>
<td>Refrig 4°C</td>
<td></td>
</tr>
<tr>
<td>Ion Analyzer</td>
<td>1000 mg/L Solution Xylene-99.9%</td>
<td>As directed</td>
<td>Stock from Source Solution</td>
<td>Refrig 4°C</td>
<td>Monthly or as directed</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Solvent Cabinet</td>
<td>Used as Received</td>
<td>N/A</td>
<td>N/A</td>
</tr>
<tr>
<td>Flashpoint Tester</td>
<td>Neat</td>
<td>As directed</td>
<td>Working Standard from stock</td>
<td>Refrig 4°C</td>
<td>Daily</td>
</tr>
<tr>
<td>Turbidimeter</td>
<td>Formazin Solution 100 NTU</td>
<td>As directed</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Conductivity Meter</td>
<td>Neat</td>
<td>RT</td>
<td>Stock Standard from Neat Compound</td>
<td>Refrig 4°C</td>
<td>Monthly</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Working Standard from Stock</td>
<td></td>
<td>Weekly</td>
</tr>
<tr>
<td>Instrument</td>
<td># Standard Initial Calibration</td>
<td>Accept/Reject Criteria-Initial</td>
<td>Frequency</td>
<td>Accept/Reject Criteria Continuing Calibration</td>
<td># Standards Continuing Calibration</td>
</tr>
<tr>
<td>-----------------------</td>
<td>--------------------------------</td>
<td>--------------------------------</td>
<td>---------------------------------------------------------------------------</td>
<td>-----------------------------------------------</td>
<td>-----------------------------------</td>
</tr>
<tr>
<td>GC (PCB/Pest)</td>
<td>3</td>
<td>%RSD. &lt;10 (608) %RSD &lt;20</td>
<td>Initial and Continuing calibration and after corrective action.</td>
<td>Conc. w/in 15% of known value</td>
<td>1</td>
</tr>
<tr>
<td></td>
<td></td>
<td>(8082,8081A)</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>GC/MS (VOA)</td>
<td>5</td>
<td>%RSD. &lt;15% (8260B) %RSD &lt;35%</td>
<td>Initial and Continuing calibration and after corrective action.</td>
<td>Conc. w/in 15% of known value</td>
<td>2</td>
</tr>
<tr>
<td></td>
<td></td>
<td>(624)</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>GC/MS (SEMI-VOL)</td>
<td>5</td>
<td>%RSD. &lt;25</td>
<td>Initial and Continuing calibration and after corrective action.</td>
<td>Conc. w/in 15% of known value</td>
<td>2</td>
</tr>
<tr>
<td>ICP</td>
<td>5</td>
<td>Linear Regression correlation coefficient &gt;0.995</td>
<td>Daily or prior to use or failure of continuing calibration</td>
<td>Conc. w/in 10% of known value</td>
<td>1</td>
</tr>
<tr>
<td>AAS Graphite Furnace</td>
<td>5</td>
<td>Linear Regression correlation coefficient &gt;0.995</td>
<td>Daily or prior to use or failure of continuing calibration</td>
<td>Conc. w/in 10% of known value</td>
<td>1</td>
</tr>
<tr>
<td>pH Meter</td>
<td>2</td>
<td>± 0.01 pH Units</td>
<td>Daily prior to use or failure of continuing calibration</td>
<td>± 0.01 pH Units</td>
<td>2</td>
</tr>
<tr>
<td>Visible Spectrophotometer</td>
<td>5</td>
<td>Linear Regression correlation coefficient of &gt;0.995</td>
<td>Daily prior to use or failure of continuing calibration</td>
<td>Conc. w/in 10% of known value</td>
<td>1</td>
</tr>
<tr>
<td>Ion Analyzer.</td>
<td>5</td>
<td>Logarithmic correlation coefficient &gt;-0.995</td>
<td>Daily Prior to use or failure of continuing calibration</td>
<td>Conc. w/in 5% of known value</td>
<td>1</td>
</tr>
<tr>
<td>Specific Conductivity Meter -</td>
<td>2</td>
<td>Conc. w/in 5% of known value (mid-range std.)</td>
<td>Daily prior to use or failure of continuing calibration</td>
<td>Conc. w/in 5% value (std. run at range of samples)</td>
<td>1</td>
</tr>
<tr>
<td>Instrument</td>
<td># Standards Initial Calibration</td>
<td>Accept/Reject Criteria-Initial</td>
<td>Frequency</td>
<td>Accept/Reject Criteria Calibration</td>
<td># Standards Continuing Calibration</td>
</tr>
<tr>
<td>-----------------------</td>
<td>---------------------------------</td>
<td>----------------------------------------------------</td>
<td>------------------------------------</td>
<td>------------------------------------</td>
<td>----------------------------------</td>
</tr>
<tr>
<td>Infrared Spectrophotometer</td>
<td>4</td>
<td>Linear Regression correlation coefficient &gt;0.995</td>
<td>Daily Prior to use or failure of continuing calibration</td>
<td>Conc. w/in 5% of known value</td>
<td>1</td>
</tr>
<tr>
<td>Analytical Balance</td>
<td>3</td>
<td>Low, mid and high range within 0.5mg of true wt.</td>
<td>Daily prior to use</td>
<td>Weight w/in 0.5mg of true wt.</td>
<td>N/A</td>
</tr>
<tr>
<td>Ovens</td>
<td>N/A</td>
<td>± 2 °C</td>
<td>Daily</td>
<td>N/A</td>
<td>N/A</td>
</tr>
<tr>
<td>Refrigerators</td>
<td>N/A</td>
<td>± 2 °C</td>
<td>Daily</td>
<td>N/A</td>
<td>N/A</td>
</tr>
<tr>
<td>Flashpoint Tester</td>
<td>1</td>
<td>± 2 °C</td>
<td>Prior to use</td>
<td>Flash w/in 2 °C of True Flash</td>
<td>1</td>
</tr>
<tr>
<td>Turbidity</td>
<td>4</td>
<td>Linear Regression correlation coefficient of &gt;0.995</td>
<td>Daily prior to use or failure of continuing calibration</td>
<td>Conc. w/in 10% of known value</td>
<td>1</td>
</tr>
</tbody>
</table>
### Table 7.4
**Standardization of Solutions**

<table>
<thead>
<tr>
<th>Analysis</th>
<th>Solution</th>
<th>Source of Primary Standard</th>
<th>Frequency of Standardization</th>
</tr>
</thead>
<tbody>
<tr>
<td>COD</td>
<td>Potassium Acid Phthalate</td>
<td>Commercial Lab Supplier</td>
<td>Daily as Needed</td>
</tr>
<tr>
<td>Chloride</td>
<td>AgNO₃ (Silver Nitrate) Titrant</td>
<td>Commercial Lab Supplier</td>
<td>Daily as Needed</td>
</tr>
<tr>
<td>Cyanide</td>
<td>KCN, Standard</td>
<td>Commercial Lab Supplier</td>
<td>Weekly</td>
</tr>
<tr>
<td>Phenols</td>
<td>Phenol Standard</td>
<td>Commercial Lab Supplier</td>
<td>Daily as Needed</td>
</tr>
<tr>
<td>Formaldehyde</td>
<td>Formaldehyde Standard.</td>
<td>Commercial Lab Supplier</td>
<td>Daily as Needed</td>
</tr>
<tr>
<td>Alkalinity</td>
<td>Sulfuric Acid</td>
<td>Commercial Lab Supplier</td>
<td>Daily as Needed</td>
</tr>
<tr>
<td>Hardness</td>
<td>Calcium and Magnesium Standards</td>
<td>Commercial Lab Supplier</td>
<td>Daily as Needed</td>
</tr>
<tr>
<td>Sulfide</td>
<td>Iodine Solution, PAO Titrant</td>
<td>Commercial Lab Supplier</td>
<td>Daily as Needed</td>
</tr>
<tr>
<td>Residual Cl</td>
<td>PAO Titrant Standard Iodine Titrant</td>
<td>Commercial Lab Supplier</td>
<td>Daily as Needed</td>
</tr>
</tbody>
</table>
8.0 **Preventive Maintenance**

8.1 **Routine Preventive Maintenance**

Analytical instrumentation has become increasingly sensitive and sophisticated in recent years and, with the increasing reliance on microprocessors, promises to become even more so in the coming years. While this development allows greater accuracy and precision, lower detection limits, and greater productivity, it also increases the modes of failure which can occur, as well as the difficulty of repairs. Thus, a program of instrument maintenance is necessary to avoid lengthy repair down-time and to ensure optimum functioning.

Preventive maintenance such as lubrication, cleaning, etc. is performed according to the procedures delineated in each instrument manual. See Table 8.1 Preventive Maintenance. Analytical balances are serviced once a year by an NIST certified balance technician. Precision and accuracy data are examined for trends and excursions beyond control limits to determine evidence of instrument malfunction. Upon discovery of a malfunction, test and corrective procedures recommended by the manufacturer's manual are implemented by the analyst. If repair cannot be effected at this stage, the laboratory director is notified and determines the appropriate action. This may involve in-house repair or a service call by a repair technician.

8.2 **Maintenance Documentation**

All malfunctions detected, as well as corrective action taken, are noted in a log book maintained with each instrument. This includes regularly scheduled preventive maintenance. All maintenance logbooks shall contain an equipment information section that shall include, laboratory identification, manufacturer's name, type identification, serial number (or other identification), date received (if available), date placed in service (if available), current location, condition when received (if available) (e.g. new, used, reconditioned), and the location of manufacturer’s instructions if available.

8.3 **Contingency Plan**

If analysis holding times will be exceeded before repairs can be affected, then samples are sent to a subcontract laboratory with specific instructions on holding times. Rush surcharges will be paid to the subcontract laboratory if sample analyses are approaching holding time limits.
<table>
<thead>
<tr>
<th>Instrument</th>
<th>Activity</th>
<th>Frequency</th>
</tr>
</thead>
<tbody>
<tr>
<td>Furnace Atomic Absorption</td>
<td>Clean furnace windows</td>
<td>Each time</td>
</tr>
<tr>
<td></td>
<td>Check plumbing connections</td>
<td>Each time</td>
</tr>
<tr>
<td></td>
<td>Clean/change graphite tube</td>
<td>Each time/Weekly (usually)</td>
</tr>
<tr>
<td></td>
<td>Check gases</td>
<td>Each time</td>
</tr>
<tr>
<td></td>
<td>Check optics</td>
<td>Each time</td>
</tr>
<tr>
<td></td>
<td>Clean/change graphite platform</td>
<td>Each time/Two weeks</td>
</tr>
<tr>
<td></td>
<td>Clean carbon tube (outside surface)</td>
<td>Each week</td>
</tr>
<tr>
<td></td>
<td>Change carbon tube acceptable</td>
<td>6 months (when As, Se run not</td>
</tr>
<tr>
<td>ICP</td>
<td>Clean plasma windows</td>
<td>Weekly</td>
</tr>
<tr>
<td></td>
<td>Check Liquid Argon</td>
<td>Daily</td>
</tr>
<tr>
<td></td>
<td>Check/change pump tubing</td>
<td>Daily/Weekly</td>
</tr>
<tr>
<td></td>
<td>Check nebulizer/change sample tips</td>
<td>Weekly/Monthly</td>
</tr>
<tr>
<td></td>
<td>Check torch/change torch</td>
<td>Daily/Monthly</td>
</tr>
<tr>
<td></td>
<td>Check waste drain</td>
<td>Daily</td>
</tr>
<tr>
<td></td>
<td>Check the tension of the pump</td>
<td>Weekly</td>
</tr>
<tr>
<td></td>
<td>Clean air filters</td>
<td>Monthly</td>
</tr>
<tr>
<td></td>
<td>Change O-rings when change torch</td>
<td>Monthly</td>
</tr>
<tr>
<td>Gas Chromatograph</td>
<td>Change liners</td>
<td>1 – 3 times per week</td>
</tr>
<tr>
<td></td>
<td>Check/change carrier gas and makeup gas</td>
<td>As necessary</td>
</tr>
<tr>
<td></td>
<td>Change carrier trap</td>
<td>When indicated</td>
</tr>
<tr>
<td></td>
<td>Change septum and O-rings</td>
<td>Weekly</td>
</tr>
<tr>
<td></td>
<td>Clip column</td>
<td>When chromatography loses sensitivity</td>
</tr>
<tr>
<td></td>
<td>Clean ECD</td>
<td>When signal reaches 150 Hz</td>
</tr>
<tr>
<td></td>
<td>Check system for gas leaks</td>
<td>Twice a year</td>
</tr>
<tr>
<td></td>
<td>ECD wipe test</td>
<td>Biweekly</td>
</tr>
<tr>
<td></td>
<td>Clean FID</td>
<td>Monthly / loss of sensitivity</td>
</tr>
<tr>
<td></td>
<td>Check flow rates</td>
<td></td>
</tr>
<tr>
<td>GC/MS-VOA</td>
<td>Check carrier gas and/or purge gas</td>
<td>Daily</td>
</tr>
<tr>
<td></td>
<td>Change carrier gas and/or purge gas</td>
<td>Tank P drops below 500 psi</td>
</tr>
<tr>
<td></td>
<td>Change in-line filters</td>
<td>(None) uses high-grade He</td>
</tr>
<tr>
<td></td>
<td>Check system for gas leaks</td>
<td>Pressure and vacuum gauges daily</td>
</tr>
<tr>
<td>VOA</td>
<td>Check Purge Trap Connection to GC</td>
<td>Daily</td>
</tr>
<tr>
<td></td>
<td>Change Trap</td>
<td>As needed</td>
</tr>
<tr>
<td>MS</td>
<td>Clean Source</td>
<td>Six months/or as needed</td>
</tr>
<tr>
<td></td>
<td>Change multiplier</td>
<td>As high voltage limit maxed</td>
</tr>
<tr>
<td></td>
<td>Change pump oil</td>
<td>Semi-annually</td>
</tr>
<tr>
<td>GC/MS-Semi-VOA</td>
<td>Check carrier gas</td>
<td>Daily</td>
</tr>
<tr>
<td></td>
<td>Change carrier gas</td>
<td>Tank P drops below 500 psi</td>
</tr>
<tr>
<td></td>
<td>Cut off end of capillary column</td>
<td>Whenever column is removed for maintenance</td>
</tr>
<tr>
<td></td>
<td>Check system for leaks</td>
<td>Gauges checked daily - instrument fully</td>
</tr>
<tr>
<td></td>
<td></td>
<td>leak checked if RTs drift</td>
</tr>
<tr>
<td>MS</td>
<td>Change Filaments</td>
<td>When they burn out</td>
</tr>
<tr>
<td></td>
<td>Clean source</td>
<td>1-2 months</td>
</tr>
<tr>
<td></td>
<td>Change multiplier</td>
<td>As it approaches high voltage limit</td>
</tr>
<tr>
<td></td>
<td>Change pump oil</td>
<td>Annually</td>
</tr>
<tr>
<td>Instrument</td>
<td>Activity</td>
<td>Frequency</td>
</tr>
<tr>
<td>-----------------------------</td>
<td>-----------------------------------------------------------------</td>
<td>-----------------------------------------------</td>
</tr>
<tr>
<td>Analytical Balances</td>
<td>Clean pan and compartment</td>
<td>Daily after use</td>
</tr>
<tr>
<td></td>
<td>Check alignment and balance</td>
<td>Daily</td>
</tr>
<tr>
<td></td>
<td>Service Balances (by Cert. Tech)</td>
<td>Annually</td>
</tr>
<tr>
<td></td>
<td>Accuracy check</td>
<td>Daily</td>
</tr>
<tr>
<td>Visible Spectrophotometer</td>
<td>Check cuvettes for scratches</td>
<td>Daily before use</td>
</tr>
<tr>
<td></td>
<td>Clean cuvettes</td>
<td>Daily before and after use</td>
</tr>
<tr>
<td></td>
<td>Instrument “zero’s”</td>
<td>Daily and after 10 samples</td>
</tr>
<tr>
<td></td>
<td>100% transmittance w/DI water</td>
<td>Daily and after 10 samples</td>
</tr>
<tr>
<td></td>
<td>Clean instrument</td>
<td>Daily and after use</td>
</tr>
<tr>
<td></td>
<td>Servicing (by Cert. Tech.)</td>
<td>Annually, calibrated with certified standards semi-annually</td>
</tr>
<tr>
<td>pH Meter</td>
<td>Electrode stored in neutral solution (pH 7)</td>
<td>Daily when not in use</td>
</tr>
<tr>
<td></td>
<td>Check connections meter electrode</td>
<td>Daily as used</td>
</tr>
<tr>
<td>Infrared Spectrophotometer</td>
<td>Check cells for scratches, dirt</td>
<td>Daily before use</td>
</tr>
<tr>
<td></td>
<td>Clean cells</td>
<td>Daily before and after use</td>
</tr>
<tr>
<td></td>
<td>Check optics</td>
<td>Semi-annually</td>
</tr>
<tr>
<td>Ion Analyzer</td>
<td>Check electrode filling solutions</td>
<td>Daily before use</td>
</tr>
<tr>
<td></td>
<td>Check connections meter electrode</td>
<td>Daily before use</td>
</tr>
<tr>
<td>Ovens</td>
<td>Clean interior and shelves</td>
<td>Weekly or as needed</td>
</tr>
<tr>
<td>Fume Hoods</td>
<td>Check air flow</td>
<td>Semi-annually by Certified Technician</td>
</tr>
<tr>
<td></td>
<td>Clean surface</td>
<td>Daily after use</td>
</tr>
<tr>
<td></td>
<td>Check vent for obstructions</td>
<td>Semi-annually by Certified Technician</td>
</tr>
<tr>
<td>Flashpoint Tester</td>
<td>Check Gas Lines</td>
<td>Daily before use</td>
</tr>
<tr>
<td></td>
<td>Check and test thermometer</td>
<td>Daily before use</td>
</tr>
<tr>
<td></td>
<td>Clean cup and gas nozzle</td>
<td>Daily after use</td>
</tr>
</tbody>
</table>
9.0 ANALYTICAL QUALITY CONTROL

Prior to acceptance and institution of any test method, GeoLabs will perform a demonstration of capability, testing the performance of the method in the available clean matrices appropriate to the method. For analytes which do not lend themselves to spiking, the demonstration of capability of will be performed using quality control samples. Results of these demonstrations will be recorded on appropriate Certification Statements and these statements as well as the supporting laboratory results will be kept on file. Such a demonstration of capability will be completed every time there is a significant change in instrument type, personnel or test method. Continuing capability is demonstrated through satisfactory analysis of laboratory control samples.

Paramount to the maintenance of reliability in analytical determinations is the need to control the accuracy and precision of analytical results. GeoLabs employs a procedure for analytical quality control which consists of analyzing check standards, duplicates, and spiked samples corresponding in number from 10% to 20% of the samples analyzed. These analyses are used to prepare control charts defining accuracy and precision. Additionally, specific routines are maintained for instrument calibration, as described in the previous section.

9.1 Laboratory QC Checks

The control tests to validate accuracy and precision include the use of:

- **Blanks (DI water and Reagent Blanks):** Frequency of 1 in batch of 20 or fewer environmental samples for organics; 1 in batch of 10 or fewer samples analyzed for inorganics and for 608 and 624 analyses.
- **Spike solutions:** same as above.
- **Duplicates:** 1 in 10 samples, per matrix or per batch analyzed for inorganics.
- **Surrogates:** Volatile, semivolatile, PCB/Pesticide and herbicide and Modified 8100, MADEP VPH and EPH analyses - all standards, blanks, samples, and spikes are dosed at the level specified in the method.
- **Laboratory Control Spike:** Semivolatile, PCB/Pesticide and Herbicide and Modified 8100 analyses - all standards, blanks, samples, and spikes are dosed at the level specified in the method.
- **Matrix Spikes:** 1 in 20 samples, per matrix/batch analyzed for organics per CMR 310.42.00. (1 in 10 for PCB/Pesticides Method 608 analysis.)
- **Matrix Spike Duplicates:** 1 in 20 samples, per matrix/batch analyzed for organics.
- **Trip Blanks:** 1 Trip blank per cooler used for transport of VOCs at client's request.
- **Internal Standards:** Volatile / semivolatile All samples, standards, blanks, spikes and duplicates are dosed at levels specified in the method.
- **Performance Standards:** PT samples will be analyzed annually, or more often, depending on regulations.
- **Continuing Calibration Standards (CCS):** Run initially, after every 10 samples, and at the end of a sample run.
- **QC Check Standards:** Run at the beginning of each run and run at a frequency of 1 in 10 samples or every 12 hours for GC/MS analysis.
- **QC Check Samples:** From ERA, run quarterly. Concentrations unknown to analysts.
- **QC Degradation Standards for Pesticides** each run when samples are run.

These operations are used systematically to determine when an analytical system is out of control and whether such a condition is due to procedural or mechanical problems.

9.2 Routine Methods to Assess Precision and Accuracy

See Table 9.1 QC Methods used to Generate Precision and Accuracy Targets.
Matrix Spike Recovery (%) = \frac{D_{ms} - D_s}{Q_{ms}} \cdot 100\%

where:  
\( D_{ms} \) = detected concentration in the Matrix Spike sample  
\( D_s \) = detected concentration in the (unspiked) sample  
\( Q_{ms} \) = Concentration of standard added to the Matrix Spike

Poor recovery in matrix spiked samples does not necessarily represent an analytical system that is out of control. It is possible that unavoidable interferences from the sample preclude efficient recoveries. Evaluation of concomitant surrogate standards can often resolve whether poor recovery is due to a matrix problem or a laboratory problem.

**Replicates / Duplicates**

Duplicate sampling analyses are performed at a rate of 1 per sample lot or every ten samples. For a pair of data points, the appropriate measure of deviation is either the percent difference:

\[ D(\%) = \frac{|d_1 - d_2|}{(d_1 + d_2)/2} \cdot 100 \]

where:  
\( D \) = Percent Difference  
\( d_1 \) = First sample value or first sample matrix spike  
\( d_2 \) = Second (duplicate) sample value or matrix spike duplicate.

or the Relative Range (R):

\[ R(\%) = \frac{d_1 - d_2}{x} \cdot 100 \text{ or } \frac{d_2 - d_1}{x} \cdot 100 \]

where \( x = \text{mean of } d_1 \text{ and } d_2 \)

In some cases multiple replicates are run on a sample and the Coefficient of Variation (or Relative Standard Deviation) would be the appropriate statistic to evaluate the dispersion of data around a mean:

\[ CV = \frac{S}{x_i} \]

where:  
\( CV \) = coefficient of variation  
\( S \) = standard deviation  
\( x_i \) = mean of i replicates (i>2)

Replicate analyses are used in one of two ways, depending on the project requirements. In the simplest case, the replicate results are reported with the other sample results along with the appropriate measure of deviation for each replicate.

**Quality Control Charts**

Quality Control Charts are used as a visual means of monitoring replicate % differences and spike recoveries. Each analytical method for which spikes are available has control chart data. Control charts are maintained within the LIMS system. New control limits are calculated based on percent recovery of the last 30 data points. If a LCS sample result falls outside the control limits (which are the average ±3 standard deviations), the analysis must be stopped until the problem has been identified and resolved. All samples analyzed with the out of control LCS sample must be reanalyzed.
Method Detection Limits

Method detection limits are determined according to procedures outlined in 40 CFR Part 136, Appendix B.
9.3 QC Requirements

If method QC requirements are more stringent than those listed, then GeoLabs will follow the QC requirements as specified and defined in the methods of interest. Line items 1-8 are understood to be minimum requirements for the determination of precision and accuracy. However, this does not imply that only the minimum QC requirements will be satisfied by GeoLabs.

<table>
<thead>
<tr>
<th>QC Method</th>
<th>Purpose</th>
<th>Conc. Level</th>
<th>Method Reference</th>
</tr>
</thead>
<tbody>
<tr>
<td>Blanks (DI Water and Reagent)</td>
<td>Monitor Contamination</td>
<td>N/A</td>
<td>All Water and Soil Methods</td>
</tr>
<tr>
<td>Trip Blanks</td>
<td>Monitor contamination during sample transport</td>
<td>N/A</td>
<td>All water and soil methods where necessary</td>
</tr>
<tr>
<td>Field Blank</td>
<td>Monitor contamination during sampling</td>
<td>N/A</td>
<td>All Water and Soil Methods where necessary</td>
</tr>
<tr>
<td>Sample Duplicates or Matrix Spike Duplicates</td>
<td>Precision</td>
<td>Low, Mid, and High Levels</td>
<td>All Water and Soil Methods</td>
</tr>
<tr>
<td>Matrix Spike (Organics/Inorganics)</td>
<td>Monitor for unusual matrix effects</td>
<td>Low Level</td>
<td>624, 8260B, 625, 8270C, 608, 8082, 8081A</td>
</tr>
<tr>
<td>Surrogates</td>
<td>Accuracy; Monitor samples and QC for processing and/or matrix problems</td>
<td>50 µg/L</td>
<td>624, 8260B</td>
</tr>
<tr>
<td></td>
<td></td>
<td>100 µg/L</td>
<td>Base/Neutrals, 625 and 8270C</td>
</tr>
<tr>
<td></td>
<td></td>
<td>150 µg/L</td>
<td>Acid Extract, 625 and 8270C</td>
</tr>
<tr>
<td></td>
<td></td>
<td>0.5 µg/mL</td>
<td>8082, 8081A, 608</td>
</tr>
<tr>
<td>QC Check Samples (EPA / ERA Samples)</td>
<td>Accuracy</td>
<td>Varies</td>
<td>Water / Wastewater Methods where necessary</td>
</tr>
<tr>
<td>Matrix Spike Duplicates (Organics)</td>
<td>Precision and monitor unusual Matrix Effects</td>
<td>Low, mid, and High Levels</td>
<td>624, 8260B, 625, 8270C, 608, 8082, 8081A</td>
</tr>
<tr>
<td>Internal Standards</td>
<td>Calculate Sample Conc. And Monitor unusual Matrix Effects</td>
<td>See Method</td>
<td>624, 8260B, 625, 8270C</td>
</tr>
<tr>
<td>Performance Standards PT Samples</td>
<td>Accuracy</td>
<td>Varies</td>
<td>All Certified Testing</td>
</tr>
<tr>
<td>Continuing Calibration Standards</td>
<td>Verify continued acceptability of initial calibration</td>
<td>Low and Mid Range</td>
<td>All Water and Soil Methods where necessary</td>
</tr>
<tr>
<td>QC Check Standards</td>
<td>Verify Accuracy of Calibration Curve</td>
<td>Low and Mid Range</td>
<td>All Water and Soil Methods where necessary</td>
</tr>
</tbody>
</table>

9.4 Actions Required When There Are QC Exceptions

Individual actions are detailed in the Method SOPs, but in general, deviations from acceptable quality control limits are treated as follows: Contamination detected in blank samples would result in the blank being re-run. Contamination in blank that also appears in samples would be cause to re-run samples as well. Inappropriate surrogate recovery would result in sample re-analysis to determine if there was a matrix interference. If failure can be demonstrated to be due to matrix interference, the analytical results would be used and the final report would make reference to the interference. Failure of batch standards (Lab Control Sample) are a cause for corrective action and the entire batch would be rerun. Data which is to be reported despite a QC exception would be annotated in the final report.
10.0 Data Reduction, Validation and Reporting

10.1 Data Reduction

Upon completion of analysis, the analyst compiles all the raw data and calculates the results in the laboratory notebook or reporting sheet for the specific analysis. All corrections must be made by crossing out, initializing and dating the correction. Associated data for calibration, standards, spikes, blanks and replicates are also assembled. Data reduction calculations are performed on standardized forms tailored to each analysis type to minimize calculation errors and to facilitate review. The individual formulas used to calculate the results are the formulas found in the SOP for each method. Temperature compensation for the conductivity meter and the pH meter is automatically adjusted by a temperature probe and associated IC circuitry. The cell constant for the conductivity meter is equal to 1, therefore, no calculations are required to achieve the analytical result. Calculations completed using internal standards for the volatile and semi-volatile organic analyses are determined by the Standard Response Factor equation found in the Hewlett Packard computer. Linear regression, logarithmic regression and single point reference calculations are performed by the analyst. The entire data package is then organized for validation.

10.2 Data Validation

The data validation audit is concerned with accuracy, precision, completeness, and understandability or legibility. All analytical documentation is examined to insure proper methods were followed and any deviations were documented. Sample log-in documentation is checked for proper holding times and preservation.

The data package is first reviewed for completeness by the Project Manager and then forwarded to the Reports Specialist. The Report Specialist also verifies that all tests and methodology requested have been performed. The Report Specialist reviews their work before submitting it to the QA department to minimize transcription errors. The QA department reviews the data package, checks that QA/QC measures are within limits, checks that transcription for accuracy and verifies selected calculations. The QA department then initials the report as being reviewed and forwards it to the Laboratory Director.

The Laboratory Director focuses primarily on whether the appropriate methods and detection limits were used and on whether all QC criteria have been satisfied, i.e. holding times have been met, QC results fall within accepted limits, sample custody was maintained. The Laboratory Director also reviews correlations among the analyses where appropriate. Documentation on method deviations and QC corrective actions are reviewed to ensure proper resolution. If the Laboratory Director suspects that proper QC has not been maintained or if the methodology has been inappropriately altered, two responses may be implemented. First, the analyst will be interviewed in an attempt to resolve any misunderstanding or omissions. Second if the situation remains unresolved, the analyst will be asked to reanalyze the samples using the proper QC and methodology. When the data package is accepted, the Laboratory Director signs it.

On the occasion when exceptional departures from documented policies and procedures are necessary, such departures are authorized by the Laboratory Director. These departures are noted on the appropriate laboratory sheet. When appropriate, reference to the departure would be made in the final report.

The Laboratory Director also reviews the data package for responsiveness of the information to the client's needs. He ensures that all of the required parameters have been examined, that field and laboratory information are in line, i.e. results for field blanks, trip blanks and any blind replicates make sense and that special instructions for methodology, accuracy and handling have been satisfied.
10.3 Data Reporting

Reports are mailed, e-mailed and/or faxed only to clients, by whatever service is suitable for their time schedule. Under severe time problems and if requested by the client, GeoLabs is willing to present the selected results by telephone to the client. However, this is done only with the client’s understanding that a validated report is only transmitted in printed form. Clients are contacted within 24 hours of validation by the Laboratory Director if data exceeds the MCL.

10.4 Data Storage

Hard copy laboratory reports as well as all associated back-up data (chromatograms, chain of custody forms, purchase orders, analysis request forms, correspondence relating to laboratory activities for the project, etc.) are filed by order number. These files are kept in the lab office for two years and are then put in storage in a on-site storage warehouse, in file boxes labeled with the order numbers, and kept for at least 8 more years. After 10 years, all information will be discarded unless other action is specifically requested by the client or contract. Laboratory notebook pages, log-in books, and instrument maintenance and calibration information are stored in the laboratory office area for 3 years and are then sent to on-site storage for an additional 7 years. Additionally, all data generated from an instrument run by a computer is stored on data cartridges for 10 years. These records are stored in storage file boxes labeled with the dates and contents.

Should GEOLABS, Inc. cease doing business for any reason, all hard copy data records would be returned to our engineering, municipal and MWRA clients. Other clients would be contacted to determine whether they wished to receive copies. If not, such copies would be destroyed. Records not subject to parsing (log books, electronic files, etc.) would be held by the owner, David Kahler, for a period of 10 years.

10.5 Data Confidentiality

Data generated by GeoLabs is confidential and is distributed only to the client, following the client’s instructions on the Chain of Custody. Requests from other interested parties are referred to the client. With permission of the client, copies of a report may be given to third parties.
11.0 Corrective Action

QC decision conditions and corrective responses vary with the types of analyses and checks being performed. See Table 10.1 for specific actions. In addition to the specifics for the QC activities described above, GeoLabs maintains a general systematic resolution procedure for any laboratory deficiency. This can be summarized as follows:

- reagents are examined for age, proper make-up, storage or use
- data are examined for calculation errors
- instruments are examined for faults and calibration
- possible sources of contamination are reviewed
- the entire analytical procedure is reviewed, by the analyst, and if unresolved, by the QA Officer for possible procedural errors

Most often, this systematic approach will resolve any QC discrepancies at one of the steps described. If, however, this fails, the analysis is rerun by re-calibrating the instrument, using newly made reagents, a different method, or if necessary by a different analyst.

The Analysts are responsible for all QC measures that are involved in their analysis methods. Analysts are trained in these procedures during their initial training. These issues are also discussed during their annual review. Most QC decisions can be made by the analysts involved, which may include instrument maintenance, and re-analysis of the sample batch. QC problems not resolved by normal procedures need to be brought to the attention of the Laboratory Director. All corrective actions must be documented in the analytical records. Samples should only be reported if all quality control measures are acceptable. If a quality control measure is found to be out of control, and the data must be reported, the analyst must fully explain the reason for the failed QC in text accompanying the effected data. The text accompanying the data with the failed QC must be transferred to the final report. Should corrective actions affect the results of any client analyses, the effected data would be corrected and the report reissued to the client.

The Laboratory Director, working with the QA Officer, is responsible for initiating and approving corrective actions. Corrective measures may be taken when results from performance evaluation samples and split samples are outside acceptance limits. The Laboratory QA Officer will look at all associated raw data and speak to the analyst and supervisor to try to determine the source of the problem. The step by step procedure outlined above would be followed.

DEP recommended corrective action will be initiated as a result of systems or performance audits, split samples or data validation review.

All deficiencies in the quality system that the quality assurance officer becomes aware of, through either internal or external audits of the laboratory, performance evaluation studies, customer complaints, etc; shall be reported to the Laboratory Director. All corrective actions to such deficiencies shall be documented on a corrective/preventive action log. Corrective actions taken will be monitored to ensure on-going compliance. Inclusion of prior corrective actions and prior deficiencies noted in external or internal audits will be included in the yearly technical review of the laboratory to monitor on-going compliance. All audit and managerial review findings will be monitored by the Laboratory Director to ensure completion within the agreed time frame.

Customer complaints are infrequent and are dealt with by trying to resolve the matter to the customer’s satisfaction. This may simply be a matter of explaining the situation and/or result or modifying our reporting format. Where a complaint raises doubt concerning the laboratory’s compliance with the laboratory’s policies
or procedures, or with the requirements of regulatory standards, including NELAC standards, or otherwise concerning the quality of the laboratory’s calibrations or tests, the laboratory ensures that those areas of activity and responsibility involved are promptly audited. Records of the complaint and subsequent action will be maintained by the Quality Assurance Manager.

<table>
<thead>
<tr>
<th>QC Activity</th>
<th>Acceptance Criteria</th>
<th>Recommended Corrective Action</th>
</tr>
</thead>
<tbody>
<tr>
<td>Initial Instrument Blank</td>
<td>Instrument Response &lt;MDL</td>
<td>Prepare another blank, if same problem, determine cause. Reagents, environment, instrument, syringe, etc.</td>
</tr>
<tr>
<td>Initial Calibration</td>
<td>Out of limits</td>
<td>Reanalyze standards and/or remake standards</td>
</tr>
<tr>
<td>QC Check Standard</td>
<td>± 5% of expected value</td>
<td>Reanalyze standard. Recalibrate and rerun samples from last good continuing calibration. check.</td>
</tr>
<tr>
<td>Method Blank</td>
<td>&lt;MDL</td>
<td>Reanalyze Blank. If still positive, determine source of contamination. Check samples for contaminants. If not present in samples check blank glassware and DI water. If present in samples, reprocess sample set</td>
</tr>
<tr>
<td>Replicates / Duplicates</td>
<td>Within lab control limits and/or method limits</td>
<td>Reanalyze sample in duplicate, as well as 1 in 3 of samples in set.</td>
</tr>
<tr>
<td>Lab Control and Matrix Spikes</td>
<td>Within lab control limits and method limits</td>
<td>Frequent failures to meet the limits for recovery or RPD warrant, investigation and correction by laboratory must be taken.</td>
</tr>
<tr>
<td>QC Check Samples</td>
<td>Within Limits Established by Manufacturer (ERA)</td>
<td>Determine cause of problem: analyst error, procedural error, etc. Re-calibrate. Re-run sample.</td>
</tr>
<tr>
<td>Surrogates</td>
<td>Within Limits Established by Method and lab control limits</td>
<td>If recovery of surrogate is out of limit range, the sample must be rerun.</td>
</tr>
<tr>
<td>Internal Standards</td>
<td>Area counts within established limits</td>
<td>Check for matrix effects, check surrogate recoveries to verify by historical data matrix effect, qualify results. If all samples effected determine source of problem, not added to samples, instrument malfunction, etc. Reanalyze sample set.</td>
</tr>
<tr>
<td>Trip Blanks</td>
<td>&lt;MDL</td>
<td>Reanalyze Blank. If still positive, determine source of contamination. Check samples for contamination. If not present in samples check blank glassware and DI water. If present in samples, reprocess sample set</td>
</tr>
<tr>
<td>Continuing Calibration</td>
<td>Within method limits</td>
<td>Maintenance if necessary. Reanalyze and remake standard if necessary.</td>
</tr>
<tr>
<td>Tuning (GC/MS)</td>
<td>Within limits established by EPA Method(s). (Every 12 hrs)</td>
<td>Maintenance and/or review of instrumental conditions. Re-analyze and remake BFB/DFTPP standard if necessary.</td>
</tr>
</tbody>
</table>
12.0 Performance and Systems Audits

12.1 Systems Audits

Managerial Review

Periodically, but at least once per year in January, the President, Lab Director, and QA Officer meet to discuss operations and problems. Topics to be discussed include: The suitability of policies and procedures; reports from managerial and supervisory personnel; the outcome of recent internal audits; corrective and preventive actions; assessment by external bodies; the results of proficiency tests; any changes in the volume and type of work undertaken; feedback from clients; complaints; other relevant factors, such as quality control activities, resources and staff training. All information presented will be reviewed by management and records of review findings and actions will be maintained by the Laboratory Director.

Internal System Audits

Beginning in January of each year, the entire laboratory is audited by the QAManager. All areas shall be audited before the end of the first quarter. Completion of the audit will be finalized with a report sent to the President and Laboratory Director for use during their yearly management review. The President will ensure that Geolabs has the proper resources necessary to perform this audit yearly.

This activity involves evaluation of all aspects of our technical operation, including:

- Analytical method employed / adherence to SOPs
- QC precision activities
- QC accuracy activities
- Instrument maintenance
- Documentation
- Traceability of standards and reagents
- Detection of improper, unethical, or illegal practices

Infractions are listed by the QA Manager on a Corrective/Preventive Action Log, with an attached detailed explanation of the finding, and discussed with the Laboratory Director. A written report is also prepared by the QA Manager and given to the appropriate personnel. If infractions are due to individual oversight, the Laboratory Director discusses the problem with the appropriate analysts. If infractions are due to a systematic error or misunderstanding, the QA Officer determines how to implement corrective action. Adoption of such modifications are reflected in an updated Standard Operating Procedure for the method affected. All corrective actions due to findings of an internal audit will be maintained in a Corrective/Preventive Action Log maintained by the Quality Assurance Manager. The QA Manager will prepare an initial report summarizing the findings of the internal audit and listing the corrective actions required and the time frame for the corrective actions to be accomplished. The QA Manager will also track all corrective/preventive actions to show that the corrective actions have been completed.

A proactive program for the detection of improper, unethical, or illegal actions will be included in our yearly technical review. This will include the audit of laboratory records, calibration data, computer back-up records, and manual integrations.
External System Audits

GeoLabs is audited by the New York Department of Health under the NELAC guidelines according to their required schedule. The MA DEP also audits GeoLabs according to their Massachusetts regulatory guidelines.

The laboratory will conduct an annual review of its quality system and its testing and calibration activities to ensure its continuing suitability and effectiveness and to introduce any necessary changes or improvements in the quality system and laboratory operations. The review will take account of reports from the Quality Assurance Officer, the outcome of recent internal audits, assessments by external bodies, the results of proficiency tests, any changes in the volume and type of work undertaken, feedback from clients, corrective actions and any other relevant factors. All information presented will be reviewed by management and records of review findings and actions will be maintained by the Laboratory Director.

12.2 Performance Audits

Internal Performance Audits

One internal performance audit is run by the analyst. The analyst checks the calibration curve for every sequence that is run. This is done by using a check standard from a source different from the standards used to generate the original calibration curve. If the check standard does not fall within the acceptable range, corrective actions are taken, e.g. necessary maintenance is done and re-calibration is performed. This procedure is documented in the lab notebook of the chemist. Any maintenance is noted in the maintenance log book for the particular instrument.

External Performance Audits

GeoLabs participates in a WP and WS Proficiency Testing Sample Program for each analyte or interdependent analyte group four times per year. The PT samples are analyzed the same as client samples, according to the detailed instructions, and following all deadlines set by the provider. Only the analyst who performs the testing on a routine basis is allowed to analyze the PT samples. Results of PT sample testing will be directed to the appropriate accrediting departments. The laboratory will not be allowed to discuss any testing results of PT samples, nor have analysis completed by another laboratory. All records of PT sample analysis and results will be kept on file with the Laboratory Director. The laboratory will not knowingly analyze a PT sample from another lab. In the instance of such a receipt of a sample, the primary accrediting authority will be notified within five business days.

In the case of a failed PT sample, a corrective action plan, detailing all corrective actions, and a schedule for further PT sample analysis to remain certified will be sent to the accrediting authority.
FIGURE 12.1

Geo Labs Internal Audit Checklist

Date of Inspection ________________________________  Department Inspected:

Inspected By ________________________________  Method Inspected:

<table>
<thead>
<tr>
<th>Deficiency</th>
<th>Analyst Audited:</th>
</tr>
</thead>
<tbody>
<tr>
<td>Equipment</td>
<td></td>
</tr>
<tr>
<td>Methodology</td>
<td></td>
</tr>
<tr>
<td>Initial Demonstration of Capability</td>
<td></td>
</tr>
<tr>
<td>Recent MDL Data</td>
<td></td>
</tr>
<tr>
<td>Precision and Accuracy Data</td>
<td></td>
</tr>
<tr>
<td>Quality Control Charts</td>
<td></td>
</tr>
<tr>
<td>QA Plan</td>
<td></td>
</tr>
<tr>
<td>SOPs</td>
<td></td>
</tr>
<tr>
<td>Documentation and Record-keeping</td>
<td></td>
</tr>
<tr>
<td>Other (specify)</td>
<td></td>
</tr>
</tbody>
</table>

Signature of Inspector ________________________________  Date:

Signature of Laboratory Director _____________________________  Date:

A copy of this report shall be furnished to analyst and Laboratory Director.

A corrective action reply within two weeks is mandatory. This must include details of corrective action performed or a detailed plan of corrective action. All supporting documentation necessary for validation of response is required.
13.0 Physical Plant

GeoLabs, Inc. is located at 45 Johnson Lane, Braintree, in approximately 12500 square feet. This space is divided into laboratory, office, sample receipt and storage, and storage locations. Laboratories are organized by function and separated to minimize adverse interactions. Hoods, safety showers, eye-wash stations and interim waste storage facilities are located as appropriate in the laboratory areas.

Deionized water is generated on site by a Millipore deionizer system. The conductance of the D.I. water is tested daily for conformance to required standards. Filters are changed as necessary.
### Appendix A  Approved NELAC Methods (See NYDOH Certificate of Approval for updated certification listings)

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Method</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aluminum</td>
<td>6010B(soil) / 200.7(pw, npw)</td>
</tr>
<tr>
<td>Antimony</td>
<td>6010B(soil) / 200.7(npw) / 200.9(pw)</td>
</tr>
<tr>
<td>Arsenic</td>
<td>6010B(soil) / 200.7(npw) / 200.9(pw)</td>
</tr>
<tr>
<td>Barium</td>
<td>6010B(soil) / 200.7(pw, npw)</td>
</tr>
<tr>
<td>Beryllium</td>
<td>6010B(soil) / 200.7(pw, npw)</td>
</tr>
<tr>
<td>Boron</td>
<td>6010B(soil) / 200.7(npw)</td>
</tr>
<tr>
<td>Cadmium</td>
<td>6010B(soil) / 200.7(npw)</td>
</tr>
<tr>
<td>Calcium</td>
<td>6010B(soil) / 200.7(pw)</td>
</tr>
<tr>
<td>Chromium</td>
<td>6010B(soil) / 200.7 (pw, npw)</td>
</tr>
<tr>
<td>Cobalt</td>
<td>6010B(soil) / 200.7(npw)</td>
</tr>
<tr>
<td>Copper</td>
<td>6010B(soil) / 200.7(pw, npw)</td>
</tr>
<tr>
<td>Iron</td>
<td>6010B(soil) / 200.7(pw, npw)</td>
</tr>
<tr>
<td>Lead</td>
<td>6010B(soil) / 200.7(npw) / 200.9(pw)</td>
</tr>
<tr>
<td>Magnesium</td>
<td>6010B(soil) / 200.7(pw, npw)</td>
</tr>
<tr>
<td>Manganese</td>
<td>6010B(soil) / 200.7(pw, npw)</td>
</tr>
<tr>
<td>Mercury</td>
<td>245.1(npw) / 7471A(soil)</td>
</tr>
<tr>
<td>Molybdenum</td>
<td>6010B(soil) / 200.7 (npw)</td>
</tr>
<tr>
<td>Nickel</td>
<td>6010B(soil) / 200.7(pw, npw)</td>
</tr>
<tr>
<td>Potassium</td>
<td>7610(soil) / 3111B SM19th ed.(pw, npw)</td>
</tr>
<tr>
<td>Selenium</td>
<td>6010B(soil) / 200.7 / 200.9(pw)</td>
</tr>
<tr>
<td>Silver</td>
<td>6010B(soil) / 200.7(pw, npw)</td>
</tr>
<tr>
<td>Sodium</td>
<td>7770(soil) / 3111B SM 19th ed. (npw)</td>
</tr>
<tr>
<td>Strontium</td>
<td>6010B(soil) / 200.7(npw)</td>
</tr>
<tr>
<td>Thallium</td>
<td>6010B(soil) / 200.7 / 200.9(pw)</td>
</tr>
<tr>
<td>Tin</td>
<td>6010B(soil) / 200.7(npw)</td>
</tr>
<tr>
<td>Titanium</td>
<td>200.7(npw)</td>
</tr>
<tr>
<td>Vanadium</td>
<td>6010B(soil) / 200.7(npw)</td>
</tr>
<tr>
<td>Zinc</td>
<td>6010B(soil) / 200.7(pw, npw)</td>
</tr>
</tbody>
</table>

**Inorganics (Non-Potable Water)**

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Method</th>
</tr>
</thead>
<tbody>
<tr>
<td>Alkalinity (Titrimetric)</td>
<td>2320B</td>
</tr>
<tr>
<td>COD</td>
<td>410.4</td>
</tr>
<tr>
<td>Chloride</td>
<td>Lachat 10-117-07-1_A</td>
</tr>
<tr>
<td>Chromium, hexavalent.</td>
<td>7196A(soil) / 3500C,r-D</td>
</tr>
<tr>
<td>Cyanide, Total</td>
<td>4500-CN-E SM 19th ed.</td>
</tr>
<tr>
<td>Cyanide, Amenable</td>
<td>4500-CN-G SM 19th ed.</td>
</tr>
<tr>
<td>Hardness</td>
<td>2340B SM 19th ed.</td>
</tr>
<tr>
<td>Hydrogen Ion (pH)</td>
<td>4500-H B SM 19th ed./ 9045C(soil)</td>
</tr>
<tr>
<td>Ignitability/Flashpoint</td>
<td>1010</td>
</tr>
<tr>
<td>MBAS</td>
<td>5540C SM 19th ed.</td>
</tr>
<tr>
<td>Kjeldahl Nitrogen- Total</td>
<td>4500-NH3 C SM 19th ed.</td>
</tr>
<tr>
<td>Nitrate</td>
<td>Lachat 10-187-04-1C</td>
</tr>
<tr>
<td>Parameter</td>
<td>Reference</td>
</tr>
<tr>
<td>----------------------------------------</td>
<td>------------------------------------</td>
</tr>
<tr>
<td>Ammonia</td>
<td>4509 NH3-C SM 19th ed.</td>
</tr>
<tr>
<td>Oil and Grease</td>
<td>1664A</td>
</tr>
<tr>
<td>Orthophosphate</td>
<td>4500 P-E SM 19th ed.</td>
</tr>
<tr>
<td>Phenols</td>
<td>420.1</td>
</tr>
<tr>
<td>Reactivity Cyanide/Sulfide</td>
<td>SW-846 Ch? Sec.7.3</td>
</tr>
<tr>
<td>Residue</td>
<td></td>
</tr>
<tr>
<td>- Total (TS)</td>
<td>2540B SM 19th ed.</td>
</tr>
<tr>
<td>- Filterable (TDS)</td>
<td>2540C SM 19th ed.</td>
</tr>
<tr>
<td>- Nonfilterable (TSS)</td>
<td>2540D SM 19th ed.</td>
</tr>
<tr>
<td>Specific Conductance</td>
<td>120.1</td>
</tr>
<tr>
<td>Sulfate</td>
<td>ASTM D516-90</td>
</tr>
<tr>
<td>Color</td>
<td>2120B SM 19th ed.</td>
</tr>
<tr>
<td>Sulfide (as S)</td>
<td>4500-S F SM 19th ed.</td>
</tr>
</tbody>
</table>

### Volatile Organic Compounds (Non-Potable Water)³⁴

<table>
<thead>
<tr>
<th>Compound</th>
<th>Reference</th>
</tr>
</thead>
<tbody>
<tr>
<td>1,1,2-Tetrachloroethane</td>
<td>8260B</td>
</tr>
<tr>
<td>1,1-Trichloroethane</td>
<td>624 / 8260B</td>
</tr>
<tr>
<td>1,1,2,2-Tetrachloroethane</td>
<td>624 / 8260B</td>
</tr>
<tr>
<td>1,1,2-Trichloroethane</td>
<td>624 / 8260B</td>
</tr>
<tr>
<td>1,1-Dichloroethane</td>
<td>624 / 8260B</td>
</tr>
<tr>
<td>1,1-Dichloroethene</td>
<td>624 / 8260B</td>
</tr>
<tr>
<td>1,1-Dichloropropene</td>
<td>8260B</td>
</tr>
<tr>
<td>1,2,3-Trichlorobenzene</td>
<td>8260B</td>
</tr>
<tr>
<td>1,2,3-Trichloropropane</td>
<td>8260B</td>
</tr>
<tr>
<td>1,2,4-Trichlorobenzene</td>
<td>8260B</td>
</tr>
<tr>
<td>1,2,4-Trimethylbenzene</td>
<td>8260B</td>
</tr>
<tr>
<td>1,2-Dibromo-3-Chloropropane</td>
<td>8260B</td>
</tr>
<tr>
<td>1,2-Dibromoethane</td>
<td>8260B</td>
</tr>
<tr>
<td>1,2-Dichlorobenzene</td>
<td>602 / 624 / 8260B</td>
</tr>
<tr>
<td>1,2-Dichloroethane</td>
<td>624 / 8260B</td>
</tr>
<tr>
<td>1,2-Dichloropropane</td>
<td>624 / 8260B</td>
</tr>
<tr>
<td>1,3,5-Trimethylbenzene</td>
<td>8260B</td>
</tr>
<tr>
<td>1,3-Dichlorobenzene</td>
<td>602 / 624 / 8260B</td>
</tr>
<tr>
<td>1,3-Dichloropropane</td>
<td>8260B</td>
</tr>
<tr>
<td>1,4-Dichlorobenzene</td>
<td>692 / 624 / 8260B</td>
</tr>
<tr>
<td>2,2-Dichloropropane</td>
<td>8260B</td>
</tr>
<tr>
<td>2-Butanone</td>
<td>8260B</td>
</tr>
<tr>
<td>2-Chloroethyl Vinyl Ether</td>
<td>624 / 8260B</td>
</tr>
<tr>
<td>2-Chlorotoluene</td>
<td>8260B</td>
</tr>
<tr>
<td>2-Hexanone</td>
<td>8260B</td>
</tr>
<tr>
<td>4-Chlorotoluene</td>
<td>8260B</td>
</tr>
<tr>
<td>4-Isopropyltoluene</td>
<td>8260B</td>
</tr>
<tr>
<td>4-Methyl-2-Pentanone</td>
<td>8260B</td>
</tr>
<tr>
<td>Acetone</td>
<td>8260B</td>
</tr>
<tr>
<td>Acrolein</td>
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</tr>
<tr>
<td>Acrylonitrile</td>
<td>624 / 8260B</td>
</tr>
<tr>
<td>Benzene</td>
<td>602 / 624 / 8260B</td>
</tr>
<tr>
<td>Volatile Organic Compounds (Solid)(^4)</td>
<td></td>
</tr>
<tr>
<td>-----------------------------------------</td>
<td></td>
</tr>
<tr>
<td>1,1,1,2-Tetrachloroethane</td>
<td>8260B</td>
</tr>
<tr>
<td>1,1,1-Trichloroethane</td>
<td>8260B</td>
</tr>
<tr>
<td>1,1,2,2-Tetrachloroethane</td>
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</tr>
<tr>
<td>1,1,2-Trichloroethane</td>
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</tr>
<tr>
<td>1,1-Dichloroethane</td>
<td>8260B</td>
</tr>
<tr>
<td>1,1-Dichloroethylene</td>
<td>8260B</td>
</tr>
<tr>
<td>1,1-Dichloroethene</td>
<td>8260B</td>
</tr>
<tr>
<td>1,1-Dichloropropene</td>
<td>8260B</td>
</tr>
<tr>
<td>1,2,3-Trichlorobenzene</td>
<td>8260B</td>
</tr>
<tr>
<td>1,2,4-Trichlorobenzene</td>
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</tr>
<tr>
<td>1,2,4-Trimethylbenzene</td>
<td>8260B</td>
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<tr>
<td>1,2-Dibromo-3-Chloropropene</td>
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<td>1,2-Dibromoethane</td>
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<td>Chemical Name</td>
<td>Code</td>
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<tr>
<td>---------------------------------------</td>
<td>----------</td>
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</tr>
<tr>
<td>1,2-Dichloroethane</td>
<td>8260B</td>
</tr>
<tr>
<td>1,2-Dichloropropane</td>
<td>8260B</td>
</tr>
<tr>
<td>1,3,5-Trimethylbenzene</td>
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<tr>
<td>1,3-Dichlorobenzene</td>
<td>8260B</td>
</tr>
<tr>
<td>1,3-Dichloropropane</td>
<td>8260B</td>
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<tr>
<td>1,4-Dichlorobenzene</td>
<td>8260B</td>
</tr>
<tr>
<td>2,2-Dichloropropane</td>
<td>8260B</td>
</tr>
<tr>
<td>2-Butanone</td>
<td>8260B</td>
</tr>
<tr>
<td>2-Chloroethyl Vinyl Ether</td>
<td>8260B</td>
</tr>
<tr>
<td>2-Chlorotoluene</td>
<td>8260B</td>
</tr>
<tr>
<td>2-Hexanone</td>
<td>8260B</td>
</tr>
<tr>
<td>4-Chlorotoluene</td>
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<td>4-Isopropyltoluene</td>
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<td>4-Methyl-2-Pentanone</td>
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<tr>
<td>Acrylonitrile</td>
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<td>Benzene</td>
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<td>Bromobenzene</td>
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<tr>
<td>Bromochloromethane</td>
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<tr>
<td>Bromodichloromethane</td>
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</tr>
<tr>
<td>Bromoform</td>
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<tr>
<td>Bromomethane</td>
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</tr>
<tr>
<td>Carbon Disulfide</td>
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<tr>
<td>Carbon Tetrachloride</td>
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<tr>
<td>Chlorobenzene</td>
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<td>Chloroethane</td>
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<td>Chloroform</td>
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<tr>
<td>cis-1,2-Dichloroethene</td>
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<td>Isopropylbenzene</td>
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<tr>
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### Pesticides (Non-Potable Water / Soil)³⁴³

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### PCBs (Non-Potable Water / Soil)³⁴³

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### Semivolatile Organic Compounds (Non-potable Water)³⁴³

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<td>2,3,4,6-Tetrachlorophenol</td>
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<td>2,4,5-Trichlorophenol</td>
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<td>4-Bromophenyl Phenyl Ether</td>
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<td>Aniline</td>
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<td>Anthracene</td>
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<tr>
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<td>Benzo(a)Pyrene</td>
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<tr>
<td>Benzo(g,h,i)Perylene</td>
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<td>Bis(2-Chloroisopropyl)Ether</td>
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<tr>
<td>Indeno(1,2,3-cd)Pyrene</td>
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<tr>
<td>Isophorone</td>
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<tr>
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**Semivolatile Organic Compounds (Solid)**

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Appendix B: Sample Preparation Table

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<td>Soil/Sediment/Sludge</td>
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<td>EP TOX Extraction</td>
<td>Soil/Solid</td>
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Method References

8. QuikChem Methods, Lachat Instruments., Milwaukee, WI.
# Appendix C: Sample Preservation and Handling

## Recommended Sample Containers, Preservation and Holding Times

<table>
<thead>
<tr>
<th>Category</th>
<th>Methods</th>
<th>Minimum Quantity</th>
<th>Recommended Container(s)</th>
<th>Required Preservation</th>
<th>Holding Time</th>
</tr>
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<tbody>
<tr>
<td><strong>Volatile Organic Analyses</strong></td>
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<tr>
<td><em>Aqueous Samples</em></td>
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<tr>
<td>Volatile Organics</td>
<td>601/602</td>
<td>40 mL</td>
<td>2 x 40 mL Glass Vials w/Teflon septum caps</td>
<td>Cool to 4°C Adjust pH &lt;2 w/HCl Remove Chlorine</td>
<td>14 Days</td>
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<tr>
<td></td>
<td>624/502.2</td>
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<td></td>
<td>524.2</td>
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<td>Acrolein &amp; Acrylonitrile</td>
<td>603</td>
<td>40 mL</td>
<td>2 x 40 mL Glass Vials w/Teflon septum caps</td>
<td>Cool to 4°C Adjust pH 4-5 w/HCl Remove Chlorine</td>
<td>14 Days</td>
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<td><strong>Solid Samples</strong></td>
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<td>Volatile Organics</td>
<td>8010/8020</td>
<td>10 g</td>
<td>1 x 125 mL Glass Vial w/Teflon septum cap</td>
<td>Cool to 4°C</td>
<td>14 Days</td>
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<tr>
<td></td>
<td>8260B</td>
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<td>Acrolein &amp; Acrylonitrile</td>
<td>8030</td>
<td>10 g</td>
<td>1 x 125 mL Glass Vial w/Teflon septum cap</td>
<td>Cool to 4°C</td>
<td>14 Days</td>
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<td><strong>Semivolatile Organics Analyses</strong></td>
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<td><em>Aqueous Samples</em></td>
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<tr>
<td>Semivolatile</td>
<td>625</td>
<td>1 L</td>
<td>2 x1L Amber Glass Bottle w/Teflon liner</td>
<td>Cool to 4°C Remove Chlorine w/ Na₂S,O₃</td>
<td>7 Days</td>
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<tr>
<td>PAHs</td>
<td>610</td>
<td>1 L</td>
<td>2 x1L Amber Glass Bottle w/Teflon liner</td>
<td>Cold to 4°C</td>
<td>7 Days</td>
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<td></td>
<td>8270C</td>
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<td>Semivolatile</td>
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<td>30 g</td>
<td>Glass Jar w/Teflon liner</td>
<td>Cool to 4°C</td>
<td>14 Days</td>
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<tr>
<td>Organics</td>
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<td>30 g</td>
<td>Glass Jar w/Teflon liner</td>
<td>Cool to 4°C</td>
<td>14 Days</td>
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<tr>
<td>PAH</td>
<td>8270C</td>
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<td>Category</td>
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<td>Recommended Container(s)</td>
<td>Required Preservation</td>
<td>Holding Time</td>
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<td>Organochlorine</td>
<td>608</td>
<td>1 L</td>
<td>2 x 1L Amber Glass Bottle w/Teflon liner</td>
<td>Cool to 4°C Adjust pH 5-9, Remove Chlorine w/ Na,S,O,</td>
<td>7 Days</td>
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<td>Pesticides &amp; PCBs</td>
<td>508</td>
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<td>2 x 1L Amber Glass Bottle w/Teflon liner</td>
<td>Remove Chlorine, w/ Na,S,O,</td>
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<td>Organophosphorus</td>
<td>614/8140</td>
<td>1 L</td>
<td>2 x 1L Amber Glass Bottle w/Teflon liner</td>
<td>Cool to 4°C Adjust pH 6-8, Remove Chlorine, w/ Na,S,O,</td>
<td>7 Days</td>
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<tr>
<td>Herbicides</td>
<td>615/8150</td>
<td>1 L</td>
<td>2 x 1L Amber Glass Bottle w/Teflon liner</td>
<td>Cool to 4°C, Remove Chlorine, w/ Na,S,O,</td>
<td>7 Days</td>
</tr>
<tr>
<td>EDB and DBCP</td>
<td>504/8011</td>
<td>40 mL</td>
<td>2 x 40 mL Glass Vials w/Teflon septum caps</td>
<td>Cool to 4°C Adjust pH &lt;2, w/HCl Remove Chlorine, w/ Na,S,O,</td>
<td>14 Days</td>
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<tr>
<td><strong>Solid Samples</strong></td>
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<td>Organochlorine</td>
<td>8081/8082</td>
<td>30 g</td>
<td>Glass Jar w/Teflon liner</td>
<td>Cool to 4°C</td>
<td>14 Days</td>
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<tr>
<td>Pesticides &amp; PCBs</td>
<td>8140</td>
<td>30 g</td>
<td>Glass Jar w/Teflon liner</td>
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<td>14 Days</td>
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<tr>
<td>Organophosphorus</td>
<td>8140</td>
<td>30 g</td>
<td>Glass Jar w/Teflon liner</td>
<td>Cool to 4°C</td>
<td>14 Days</td>
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<tr>
<td>Herbicides</td>
<td>8150</td>
<td>30 g</td>
<td>Glass Jar w/Teflon liner</td>
<td>Cool to 4°C</td>
<td>14 Days</td>
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<tr>
<td>EDB and DBCP</td>
<td>8260B</td>
<td>10 g</td>
<td>1 x 125 mL Glass Vial w/Teflon septa cap</td>
<td>Cool to 4°C</td>
<td>14 Days</td>
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<td><strong>Hydrocarbon Analyses</strong></td>
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<tr>
<td>Hydrocarbon</td>
<td>8100 Modified</td>
<td>1 L</td>
<td>2 x 1L Amber Glass Bottle w/Teflon liner</td>
<td>Cool to 4°C Adjust pH &lt;2 w/H,SO,</td>
<td>7 Days</td>
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<td>Fingerprint (GC/FID)</td>
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<td>TPH/DRO</td>
<td>418.1/8100M</td>
<td>1 L</td>
<td>2 x 1L Amber Glass Bottle w/Teflon liner</td>
<td>Adjust pH &lt;2 w/H,SO,</td>
<td>28 Days</td>
</tr>
<tr>
<td></td>
<td>8100</td>
<td></td>
<td></td>
<td>Cool to 4°C Adjust pH &lt;2 w/H,SO,</td>
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<tr>
<td>Oil and Grease</td>
<td>1664A</td>
<td>1 L</td>
<td>2 x 1L Amber Glass Bottle w/Teflon liner</td>
<td>Cool to 4°C Adjust pH &lt;2 w/H,SO, or HCl</td>
<td>28 Days</td>
</tr>
<tr>
<td>Gasoline Range</td>
<td>8015</td>
<td>40 mL</td>
<td>2 x 40 mL Glass Vials w/Teflon septa caps</td>
<td>Adjust pH &lt;2 w/HCl</td>
<td>14 Days</td>
</tr>
<tr>
<td>Category</td>
<td>Methods</td>
<td>Minimum Quantity</td>
<td>Recommended Container(s)</td>
<td>Required Preservation</td>
<td>Holding Time</td>
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<td>Solid Samples</td>
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</tr>
<tr>
<td>Hydrocarbon</td>
<td>8100 Modified</td>
<td>30 g</td>
<td>Glass Jar w/Teflon liner</td>
<td>Cool to 4°C</td>
<td>14 Days</td>
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<tr>
<td>Fingerprint (GC/FID)</td>
<td>418.1/8100M</td>
<td>30 g</td>
<td>Glass Jar w/Teflon liner</td>
<td>Cool to 4°C</td>
<td>14 Days</td>
</tr>
<tr>
<td>TPH/DRO</td>
<td>1664A</td>
<td>30 g</td>
<td>Glass Jar w/Teflon liner</td>
<td>Cool to 4°C</td>
<td>14 Days</td>
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<tr>
<td>Oil and Grease</td>
<td>8015</td>
<td>10 g</td>
<td>1 x 125 mL Glass Vial w/Teflon septa cap</td>
<td>Cool to 4°C</td>
<td>14 Days</td>
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<tr>
<td>Gasoline Range Organics</td>
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**Metals Analyses**

**Aqueous Samples**

<table>
<thead>
<tr>
<th>Category</th>
<th>Methods</th>
<th>Minimum Quantity</th>
<th>Recommended Container(s)</th>
<th>Required Preservation</th>
<th>Holding Time</th>
</tr>
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<tbody>
<tr>
<td>Total Metals (except Mercury)</td>
<td>200.7/200.9</td>
<td>100 mL</td>
<td>Plastic Bottle</td>
<td>Cool to 4°C</td>
<td>180 Days</td>
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<tr>
<td>Dissolved Metals (except Mercury)</td>
<td>6010B/7000’s</td>
<td>100 mL</td>
<td>Plastic Bottle</td>
<td>Adjust pH to &lt;2 w/HNO₃</td>
<td>180 Days</td>
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<tr>
<td>Total Mercury</td>
<td>245.1</td>
<td>100 mL</td>
<td>Plastic Bottle</td>
<td>Filter First Cool to 4°C</td>
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<td></td>
<td>7470A</td>
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<tr>
<td>Dissolved Mercury</td>
<td>245.1</td>
<td>100 mL</td>
<td>Plastic Bottle</td>
<td>Adjust pH to &lt;2 w/HNO₃</td>
<td>28 Days</td>
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<tr>
<td></td>
<td>7470A</td>
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<td>Filter First Cool to 4°C</td>
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<tr>
<td>Drinking Water</td>
<td>200.9</td>
<td>1 L</td>
<td>1 x 1 L Plastic Bottle</td>
<td>Adjust pH &lt;2 w/HNO₃</td>
<td>180 Days</td>
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<tr>
<td>Lead &amp; Copper Rule</td>
<td>200.7</td>
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<td>Cool to 4°C</td>
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<tr>
<td>Chromium, Hexavalent</td>
<td>218.1-5</td>
<td>200 mL</td>
<td>Plastic Bottle</td>
<td>Cool to 4°C</td>
<td>24 Hours</td>
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**Solid Samples**

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<th>Required Preservation</th>
<th>Holding Time</th>
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<td>Total Metals (except Mercury)</td>
<td>6010B</td>
<td>2 g</td>
<td>Glass Jar w/Teflon liner</td>
<td>Cool to 4°C</td>
<td>180 Days</td>
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<tr>
<td>Total Mercury</td>
<td>7471</td>
<td>0.6 g</td>
<td>Glass Jar w/Teflon liner</td>
<td>Cool to 4°C</td>
<td>28 Days</td>
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<td>Category</td>
<td>Methods</td>
<td>Minimum Quantity</td>
<td>Recommended Container(s)</td>
<td>Required Preservation</td>
<td>Holding Time</td>
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<td>Solid Samples</td>
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<td>TCLP Volatile</td>
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<td>150 g</td>
<td>2 x 125 mL Glass</td>
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<td>8260B</td>
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<td>Vial w/Teflon septum cap</td>
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<td>TCLP Metals, SEMI-Volatiles, Pesticides and Herbicides</td>
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<td>300 g</td>
<td>Glass Jar</td>
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<td>28 Days</td>
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<td>7000's/8081A</td>
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<td>w/Teflon liner</td>
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<td>Ignitability</td>
<td>1010 Modified</td>
<td>100 g</td>
<td>Plastic or Glass Jar</td>
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<td>Corrosivity</td>
<td>9045</td>
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<td>Plastic or Glass Jar</td>
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<td>Reactivity</td>
<td>SW-846</td>
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<td>Paint Filter</td>
<td>9095</td>
<td>100 g</td>
<td>Plastic or Glass Jar</td>
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<td>Color</td>
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<td>Conductance</td>
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<td>Plastic or Glass Bottle</td>
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<td>28 Days</td>
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<td>Hardness</td>
<td>130.1-2</td>
<td>100 mL</td>
<td>Plastic or Glass Bottle</td>
<td>Cool to 4°C</td>
<td>180 Days</td>
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<td>Odor</td>
<td>140.0</td>
<td>200 mL</td>
<td>Glass Bottle Only</td>
<td>Adjust pH&lt;2 w/HNO₃</td>
<td>24 Hours</td>
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<td>pH</td>
<td>4500-H-B</td>
<td>25 mL</td>
<td>Plastic or Glass Bottle</td>
<td>Cool to 4°C</td>
<td>None</td>
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<td>Solids, Total</td>
<td>2540C</td>
<td>100 mL</td>
<td>Plastic or Glass Bottle</td>
<td>Cool to 4°C</td>
<td>Immediately</td>
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<tr>
<td>Dissolved (TDS)</td>
<td>2540D</td>
<td>100 mL</td>
<td>Plastic or Glass Bottle</td>
<td>Cool to 4°C</td>
<td>7 Days</td>
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<td>Solids, Total (TSS)</td>
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<td>Solids, Total (TS)</td>
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<td>100 mL</td>
<td>Plastic or Glass Bottle</td>
<td>Cool to 4°C</td>
<td>7 Days</td>
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<tr>
<td>Solids, Total</td>
<td>160.4</td>
<td>100 mL</td>
<td>Plastic or Glass Bottle</td>
<td>Cool to 4°C</td>
<td>7 Days</td>
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<td>Solids, Settleable (SS)</td>
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<td>100 mL</td>
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<td>48 Hours</td>
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<tr>
<td>Turbidity</td>
<td>180.1</td>
<td>100 mL</td>
<td>Plastic or Glass Bottle</td>
<td>Cool to 4°C</td>
<td>48 Hours</td>
</tr>
<tr>
<td>Category</td>
<td>Methods</td>
<td>Minimum Quantity</td>
<td>Recommended Container(s)</td>
<td>Required Preservation</td>
<td>Holding Time</td>
</tr>
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<td>Aqueous Samples</td>
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</tr>
<tr>
<td>Acidity</td>
<td>305.1</td>
<td>100 mL</td>
<td>Plastic or Glass Bottle</td>
<td>Cool to 4°C</td>
<td>14 Days</td>
</tr>
<tr>
<td>Alkalinity</td>
<td>2320B</td>
<td>100 mL</td>
<td>Plastic or Glass Bottle</td>
<td>Cool to 4°C</td>
<td>14 Days</td>
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<tr>
<td>Bromide</td>
<td>320.1</td>
<td>100 mL</td>
<td>Plastic or Glass Bottle</td>
<td>None</td>
<td>28 Days</td>
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<tr>
<td>Chloride</td>
<td>L10-117-07-1 A</td>
<td>50 mL</td>
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<td>None</td>
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<td>Chlorine, Total Residual Cyanide</td>
<td>330.1-5</td>
<td>200 mL</td>
<td>Plastic or Glass Bottle</td>
<td>None</td>
<td>Immediately</td>
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<tr>
<td>Fluoride</td>
<td>340.1-3</td>
<td>300 mL</td>
<td>Plastic Bottle Only</td>
<td>Cool to 4°C Adj pH &gt;12 w/NaOH Remove Sulfide</td>
<td>14 Days</td>
</tr>
<tr>
<td>Iodine</td>
<td>345.1</td>
<td>100 mL</td>
<td>Plastic or Glass Bottle</td>
<td>None</td>
<td>28 Days</td>
</tr>
<tr>
<td>Nitrogen, Ammonia</td>
<td>4500-NH3 C</td>
<td>400 mL</td>
<td>Plastic or Glass Bottle</td>
<td>Cool to 4°C Adj pH &lt;2 w/H2SO3</td>
<td>28 Days</td>
</tr>
<tr>
<td>Nitrogen, Total Kjeldahl</td>
<td>4500-NH3 C</td>
<td>500 mL</td>
<td>Plastic or Glass Bottle</td>
<td>Cool to 4°C Adj pH &lt;2 w/H2SO3</td>
<td>28 Days</td>
</tr>
<tr>
<td>Nitrate plus Nitrite</td>
<td>353.1-3</td>
<td>100 mL</td>
<td>Plastic or Glass Bottle</td>
<td>Cool to 4°C Adj pH &lt;2 w/H2SO3</td>
<td>28 Days</td>
</tr>
<tr>
<td>Nitrogen, Nitrate</td>
<td>L10-107-04-1-C</td>
<td>100 mL</td>
<td>Plastic or Glass Bottle</td>
<td>Cool to 4°C</td>
<td>48 Hours</td>
</tr>
<tr>
<td>Nitrogen, Nitrite</td>
<td>354.1</td>
<td>50 mL</td>
<td>Plastic or Glass Bottle</td>
<td>Cool to 4°C</td>
<td>48 Hours</td>
</tr>
<tr>
<td>Orthophosphate</td>
<td>4500P-E</td>
<td>50 mL</td>
<td>Glass Bottle Only</td>
<td>Filer (0.45um) Cool to 4°C</td>
<td>48 Hours</td>
</tr>
<tr>
<td>Oxygen, Dissolved Phosphorous, Total</td>
<td>360.1</td>
<td>300 mL</td>
<td>Glass Bottle Only</td>
<td>None</td>
<td>Immediately</td>
</tr>
<tr>
<td>Silica</td>
<td>370.1</td>
<td>50 mL</td>
<td>Plastic Bottle Only</td>
<td>Cool to 4°C</td>
<td>28 Days</td>
</tr>
<tr>
<td>Sulfate</td>
<td>D516-90</td>
<td>50 mL</td>
<td>Plastic or Glass Bottle</td>
<td>Cool to 4°C</td>
<td>28 Days</td>
</tr>
<tr>
<td>Sulfide</td>
<td>4500-S-F</td>
<td>500 mL</td>
<td>Plastic or Glass Bottle</td>
<td>Cool to 4°C Adj pH &gt;9 w/ NaOH, 2 mL Zinc Acetate</td>
<td>7 Days</td>
</tr>
<tr>
<td>Category</td>
<td>Methods</td>
<td>Minimum Quantity</td>
<td>Recommended Container(s)</td>
<td>Required Preservation</td>
<td>Holding Time</td>
</tr>
<tr>
<td>-------------------------</td>
<td>---------</td>
<td>------------------</td>
<td>--------------------------</td>
<td>-----------------------</td>
<td>--------------</td>
</tr>
<tr>
<td><strong>Conventional Organic Analyses</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Aqueous Samples</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Biochemical</td>
<td>405.1</td>
<td>1 L</td>
<td>Plastic or Glass Bottle</td>
<td>Cool to 4°C</td>
<td>48 Hours</td>
</tr>
<tr>
<td>Oxygen Demand (5 Day BOD)</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Chemical Oxygen Demand (COD)</td>
<td>410.1-4</td>
<td>50 mL</td>
<td>Plastic or Glass Bottle</td>
<td>Cool to 4°C Adjust pH &lt;2 w/ H&lt;sub&gt;2&lt;/sub&gt;SO&lt;sub&gt;4&lt;/sub&gt;, Cool to 4°C Adjust pH &lt;2 w/ H&lt;sub&gt;2&lt;/sub&gt;SO&lt;sub&gt;4&lt;/sub&gt;,</td>
<td>28 Days</td>
</tr>
<tr>
<td>Organic Carbon. Total (TOC)</td>
<td>415.1</td>
<td>25 mL</td>
<td>Plastic or Glass Bottle</td>
<td>Cool to 4°C Adjust pH &lt;2 w/ H&lt;sub&gt;2&lt;/sub&gt;SO&lt;sub&gt;4&lt;/sub&gt;, Cool to 4°C Adjust pH &lt;2 w/ H&lt;sub&gt;2&lt;/sub&gt;SO&lt;sub&gt;4&lt;/sub&gt;,</td>
<td>28 Days</td>
</tr>
<tr>
<td>Phenolics</td>
<td>420.1-3</td>
<td>500 mL</td>
<td>Glass Bottle Only</td>
<td>Cool to 4°C</td>
<td>28 Days</td>
</tr>
<tr>
<td>Surfactants (MBAS)</td>
<td>5540C</td>
<td>250 mL</td>
<td>Plastic or Glass Bottle</td>
<td>Cool to 4°C</td>
<td>48 Hours</td>
</tr>
<tr>
<td><strong>Bacterial Analyses</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Aqueous Samples</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Coliform</td>
<td></td>
<td>100 mL</td>
<td>Whirlback Bag OR</td>
<td>Cool to 4°C</td>
<td>6 Hours</td>
</tr>
<tr>
<td>Total and Fecal</td>
<td></td>
<td></td>
<td>Sterilized Bottle</td>
<td>Remove Chlorine w/ Na&lt;sub&gt;2&lt;/sub&gt;S</td>
<td></td>
</tr>
</tbody>
</table>
Appendix D: Certifications and accreditations

GeoLabs Inc. maintains certifications or accreditations with numerous states. At the time of this QA Plan revision, the laboratory has accreditations or certifications with the following organizations:

<table>
<thead>
<tr>
<th>Organization</th>
<th>Certificate Number</th>
</tr>
</thead>
<tbody>
<tr>
<td>Massachusetts DEP</td>
<td>M-MA-015</td>
</tr>
<tr>
<td>New York DOH NELAP</td>
<td>11796</td>
</tr>
<tr>
<td>Pennsylvania DEP NELAP</td>
<td>68-03417</td>
</tr>
<tr>
<td>New Jersey DEP NELAP</td>
<td>MA-009</td>
</tr>
<tr>
<td>Connecticut DPH</td>
<td>PH-0148</td>
</tr>
<tr>
<td>Rhode Island DOH</td>
<td>LA000252</td>
</tr>
<tr>
<td>Maine DHHS</td>
<td>2010018</td>
</tr>
<tr>
<td>New Hampshire ELAP-NELAP</td>
<td>250809</td>
</tr>
</tbody>
</table>

The certificates and parameter lists for each state may be found on the GeoLabs website (www.geolabs.com) and in the QA office.
GEOLABS, Inc.
Standard Operating Procedure

INDUCTIVELY COUPLED PLASMA-ATOMIC EMISSION SPECTROMETRY
Revision: 3.0

EPA Method 6010B

Effective Date 3/04/2009

Approved by:

Mark Perkins; Quality Assurance Manager

Jim Chen; Laboratory Director
STANDARD OPERATING PROCEDURE

Inductively Coupled Plasma-Atomic Emission Spectrometry
EPA Method 6010B

1.0 Scope and Application

1.1 Inductively coupled plasma-atomic emission spectrometry (ICP-AES) determines metals in solution. All matrices require digestion prior to analysis.

1.2 This method may be used for the following analytes:

- Aluminum
- Antimony
- Arsenic
- Barium
- Beryllium
- Boron
- Cadmium
- Calcium
- Chromium
- Cobalt
- Copper
- Iron
- Lead
- Lithium
- Magnesium
- Manganese
- Molybdenum
- Nickel
- Potassium
- Selenium
- Silver
- Sodium
- Strontium
- Thallium
- Tin
- Titanium
- Vanadium
- Zinc

2.0 Summary of Method

2.1 The analysis involves multi-element determination using ICP-AES. The instrument measures characteristic emission spectra by optical spectrometry. Samples are nebulized and the resulting aerosol is transported to the plasma torch. Emissions are produced by radio-frequency inductively coupled plasma.

3.0 Modifications

3.1 The digestion procedure has been modified to conform to the EPA 3010A Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by ICP Spectroscopy. The sample and reagent volumes listed in this method have been reduced by half to speed the digestion process and reduce the amount of waste produced.

4.0 Definitions

4.1 Calibration Blank: A volume of reagent water acidified with the same acid matrix as in the calibration standards. The calibration blank is a zero standard.

4.2 Method Blank: A volume of reagent water that is digested exactly as a sample. The method blank is used to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus.

4.3 Laboratory Control Sample: A volume of reagent water to which known amounts of method analytes are added. The LCS is analyzed exactly like a sample, and its purpose is to determine the laboratory accuracy for the method.
4.4 Matrix Spike Sample: A volume of an environmental sample to which known amounts of method analytes are added. The MS is analyzed exactly like a sample and its purpose is to determine whether the sample matrix contributes bias to the analytical results. The MS is normally analyzed in duplicate (MS/MSD).

4.5 Calibration Standard: A solution prepared from the dilution of stock standard solutions. The CAL solns. Are used to calibrate the instrument.

4.6 Cal. Check: A solution with a known amount of analyte, which is run after every 10 samples to check the calibration of the instrument.

4.7 Stock Standard Solution: A 1000-ppm standard from which the CAL STD's are diluted.

5.0 Interferences

5.1 Spectral interferences are caused by background emission from continuous or recombination phenomena, stray light from the line emission of high concentration elements, overlap of a spectral line from another element, or unresolved overlap of molecular band spectra.

5.2 Physical interferences are effects associated with the sample nebulization and transport processes. Changes in viscosity and surface tension can cause significant inaccuracies, especially in samples containing high dissolved solids or high acid concentrations. If physical interferences are present, they must be reduced by diluting the sample or by using a peristaltic pump. Another problem that can occur with high dissolved solids is salt buildup at the tip of the nebulizer, affecting aerosol flow rate and causing instrumental drift. Diluting the sample can control the problem. Also, it has been reported that better control of the argon flow rate, especially to the nebulizer, improves instrument performance.

5.3 Chemical interferences include molecular compound formation, ionization effects, and solute vaporization effects. Normally, these effects are not significant with the ICP technique, but if observed, can be minimized by careful selection of operating conditions (incident power, observation position, and so forth), by buffering of the sample, by matrix matching, and by standard addition procedures. Chemical interferences are highly dependent on matrix type and the specific analyte element.

5.4 Memory interferences result when analytes in a previous sample contribute to the signals measured in a new sample. Memory effects can result from sample deposition on the uptake tubing to the nebulizer and from the build up of sample material in the plasma torch and spray chamber. The site where these effects occur is dependent on the element and can be minimized by flushing the system with a rinse blank between samples. The possibility of memory interferences should be recognized within an analytical run and suitable rinse times should be used to reduce them. The rinse times necessary for a particular element must be estimated prior to analysis. This may be achieved by aspirating a standard containing elements at a concentration ten times the usual amount or at the top of the linear dynamic range. The aspiration time for this sample should be the same as a normal sample analysis period, followed by analysis of the rinse blank at designated intervals. The length of time required to reduce analyte signals to within a factor of two of the method detection limit should be noted. Until the required rinse time is established, this method suggests a rinse period of at least 60 seconds between samples.
and standards. If memory interference is suspected, the sample must be reanalyzed after a rinse period of sufficient length.

5.5 Users are advised that high salt concentrations can cause analyte signal suppressions.

6.0 Safety

6.1 See GeoLabs Chemical Hygiene Plan for information on laboratory safety.

7.0 Apparatus and Materials

7.1 Inductively coupled plasma emission spectrometer (ICP)

7.1.1 Computer-controlled emission spectrometer with background-correction capability.

7.1.2 Radio-frequency generator compliant with FCC regulations.

7.1.3 Argon gas supply – High purity grade (99.99%).

7.1.4 A variable speed peristaltic pump is used to deliver both standard and sample solutions to the nebulizer.

7.2 Assorted Class A volumetric flasks, pipettes, beakers and ribbed watch glasses

7.3 A temperature adjusted hot plate capable of maintaining a temp of 95 degrees C

7.4 An air displacement pipetter for spiking purposes

8.0 Reagents and Standards

8.1 De-Ionized water (DI)

8.2 Nitric Acid (concentrated), HNO3

8.2.1 5% HNO3

8.3 Hydrochloric Acid (concentrated), HCL

8.3.1 (1+1) HCL

8.4 Standard stock solutions (purchased), single element ICP standards from 99.99 to 99.999% pure from two sources. Multi-element (21 element) standard and 7A standard (silver barium boron aluminum potassium and sodium)

9.0 Sample Collection, Preservation, and Handling

9.1 Prior to the collection of an aqueous sample, consideration should be given to the type of data required, (i.e., dissolved or total recoverable), so that appropriate preservation and pretreatment steps can be taken. A sample cannot be filtered that has already been acidified. The pH of all aqueous samples must be tested at the time of receipt by the laboratory log-in staff. If the sample is properly acid preserved it may be held for up to 6 months before analysis.
9.2 For the determination of the dissolved elements, the sample must be filtered through a 0.45 micron pore diameter membrane filter at the time of collection or as soon thereafter as practically possible. The laboratory will filter clients samples if a request is made, as clearly noted on the chain of custody. Acidify the filtrate with (i+1) nitric acid immediately following filtration to pH<2.

9.3 For the determination of total recoverable elements in aqueous samples, samples are not filtered, but acidified with (i+1) nitric acid to pH<2. Samples should be received within 2 weeks of collection if client desires laboratory to perform preservation. Following acidification, the sample should be mixed, held for 16 hours, and then verified to be pH<2, just prior to withdrawing an aliquot for processing. If for some reason such as high alkalinity the sample pH is verified to be >2, more acid must be added and the sample held for 16 hours until verified to be pH <2.

10.0 Digestion Procedure

10.1 Transfer a 50-ml aliquot of well-mixed sample to a beaker.

10.2 Add 1.5 ml of concentrated nitric acid to the beaker and sample and cover with a ribbed watch glass. Place the beaker on the hot plate at approximately but no higher than 85°C and evaporate to a low volume (approximately 20 ml), making certain that no portion of the bottom of the beaker is allowed to go dry.

10.3 Cool the beaker and add another 1.5 ml of conc. HNO₃, and return to the hot plate. Increase the temperature of the hot plate so that a gentle reflux action occurs. Vigorous boiling must be avoided.

NOTE: If a sample is allowed to go to dryness, low recoveries will result. Should this occur, discard the sample and re-digest.

10.4 Cool the beaker, add 5 ml of 1:1 HCl and cover the beaker and reflux for 15 minutes to dissolve any precipitate or residue.

10.5 Wash down the beaker walls and watch glass with DI water and, if necessary, filter the sample. Adjust the final volume to 50 ml.

NOTE: The sample and reagent volumes listed in the source method (EPA 3010A) were all reduced by half. This was done to reduce the overall digestion time and reduce waste.

11.0 Analysis Procedure

11.1 Inspect the sample introduction system. Clean or replace as needed.

11.2 Check instrument operating conditions.

11.2.1 ICP OPERATING CONDITIONS

<table>
<thead>
<tr>
<th>PARAMETER</th>
<th>RADIAL TORCH AXIAL TORCH</th>
</tr>
</thead>
<tbody>
<tr>
<td>Power, kW</td>
<td>1.3-1.5</td>
</tr>
<tr>
<td>Coolant Flow, L/min</td>
<td>14-18</td>
</tr>
<tr>
<td>Aux. Flow, L/min</td>
<td>0-1.5</td>
</tr>
<tr>
<td>Neb L/min</td>
<td>0.5-0.8</td>
</tr>
<tr>
<td>Uptake Rate, ml/min</td>
<td>1.5</td>
</tr>
</tbody>
</table>

1.5
11.3 The instrument must be thermally stable before calibration and analysis. After igniting the torch, allow the instrument to warm-up for 30-40 minutes.

11.4 For initial and daily operation, calibrate the instrument to the manufacturer's recommended conditions, using mixed calibration standards and the calibration blank. A peristaltic pump must be used to introduce all solutions to the nebulizer. All solutions should be aspirated for a minimum of 60 seconds. The curve for each wavelength must have a correlation coefficient of 0.995 or higher.

11.5 Samples should be analyzed in the same manner used for the calibration of the instrument.

12.0 Data Analysis

12.1 The sample data should be reported in units of mg/L for aqueous samples and mg/Kg for solids.

12.2 Convert mg/L to mg/Kg by dividing the result by (weight x % solids) and then multiply that result by 100 ml.

12.3 Do not report data below the determined analyte MDL.

12.4 Report analyte concentrations up to three significant figures.

12.5 IPC must be within +/- 10% of the true value initially and +/-10% of the true value thereafter.

12.6 All blanks must be < Or = the MDL.

13.0 Quality Control

13.1 Each analyst using this method is required to follow the formal quality control program as described in this SOP and the current version of the GEOLABS QA PLAN. The requirements of this program include the demonstration of capability, and the periodic analysis of method blanks and laboratory control samples (LCS). The analyst is required to maintain and use performance records in the form of control charts, to define the quality of the data that is generated, for the purpose of acceptance.

13.2 Initial Demonstration of Performance (mandatory)

13.2.1 The initial demonstration of performance is used to characterize instrument performance (determination of linear dynamic ranges and analysis of quality control samples) and laboratory performance (determination of method detection limits) prior to analyses conducted by this method.

13.2.2 Linear dynamic range (LDR)- The upper limit of the LDR must be established for each wavelength utilized. It must be determined from a linear calibration prepared in the normal manner using the established analytical operating procedure for the instrument. The LDR should be determined by analyzing exceedingly higher standard concentrations of the analyte until the observed analyte concentration is no more than 10% below the stated concentration of the standard. Determined LDRs must be documented and kept on file. The LDR which may be used for the analysis of samples should be judged by the analyst
from the resulting data. Determined sample analyte concentrations that are greater than 90% of the determined upper LDR limit must be diluted and reanalyzed. The LDRs should be verified annually or whenever, in the judgment of the analyst, a change in analytical performance caused by either a change in instrument hardware or operating conditions would dictate they be re-determined.

13.2.3 Quality control sample (QCS)- When beginning the use of this method, on a quarterly basis, after the preparation of stock or calibration standard solutions or as required to meet data-quality needs, verify the calibration standards and acceptable instrument performance with the preparation and analyses of a QCS. To verify the calibration standards the determined mean concentrations from three analyses of the QCS must be within ±5% of the stated values. If the calibration standard cannot be verified, performance of the determinative step of the method is unacceptable. The source of the problem must be identified and corrected before either proceeding on with the initial determination of method detection limits or continuing with on-going analyses.

13.2.4 Method Detection Limit (MDL) - MDLs must be established for all wavelengths utilized, using reagent water (blank) fortified at a concentration of two to three times the estimated instrument detection limit. To determine MDL values, take seven replicate aliquots of the fortified reagent water and process through the entire analytical method. Perform all calculations defined in the method and report the concentration values in the appropriate units. Calculate the MDL as follows:

\[
\text{MDL} = (t) \times (S)
\]

Where:
- \( t \) = student's \( t \) value for a 99% confidence level and a standard deviation estimate with \( n-1 \) degrees of freedom (\( t = 3.14 \) for seven replicates)
- \( S \) = standard deviation of the replicate analyses

The MDLs must be sufficient to detect analytes at the required levels according to compliance monitoring regulation. MDLs should be determined annually, when a new operator begins work or whenever, in the judgment of the analyst, a change in analytical performance caused by either a change in instrument hardware or operating conditions would dictate they be re-determined.

13.3 Method Blanks (MB)- The laboratory must analyze at least one MB with each batch of 20 or fewer samples of the same matrix. MB data are used to assess contamination from the laboratory environment. MB values that exceed the MDL indicate laboratory or reagent contamination should be suspected. When MB values constitute 10% or more of the analyte level determined for a sample or is 2.2 times the analyte MDL whichever is greater, fresh aliquots of the samples must be prepared and analyzed again for the affected analytes after the source of contamination has been corrected and acceptable MB values have been obtained.
13.4 **Laboratory Control Sample (LCS)** - The laboratory must analyze at least one LCS with each batch of samples. Calculate accuracy as percent recovery using the following equation:

\[ R = \frac{\text{LCS} - \text{MB}}{\text{S}} \times 100 \]

Where:
- \( R \) = percent recovery
- LCS = Lab control sample
- MB = Method Blank
- S = concentration equivalent of analyte added to fortify the LCS solution

If the recovery of any analyte falls outside the required control limits of 85-115%, that analyte is judged out of control, and the source of the problem should be identified and resolved before continuing analyses. Control chart limits developed from the mean percent recovery and the standard deviation of the mean percent recovery shall be updated monthly using the most recent 30 data points. The upper and lower control limits used shall be within both the method required control limits of 85-115% and the analysts control chart limits.

13.5 **Matrix Spike (MS)** - Shall be performed at a frequency of one in 10 samples per matrix type per sample preparation method. The selected sample shall be rotated among client samples so that various matrix problems may be noted and/or addressed. Poor performance in a matrix spike may indicate a problem with the sample composition and shall be reported to the client whose sample was used for the spike.

13.6 **Matrix Spike Duplicate (MSD) or Sample Duplicates** - shall be analyzed at a minimum of 1 in 10 samples per sample preparation method. Duplicate results are used to establish control chart limits for precision. Results of duplicates (\%RPD) are to be compared to control chart limits for replicate analysis to determine acceptable reproducibility.

13.7 **Instrument Performance Check (IPC) solution** - The IPC solution is used to periodically verify instrument performance during analysis. It should be prepared in the same acid mixture as the calibration standards. Silver must be limited to <0.5 mg/L; while potassium because of higher MDLs and silica because of potential contamination should be at concentrations of 10 mg/L. For other analytes a concentration of 2 mg/L is recommended. The IPC solution must be prepared from the same standard stock solutions used to prepare the calibration standards and stored in an FEP bottle. For all determinations the laboratory must analyze the IPC solution and a calibration blank immediately following daily calibration, after every 10th sample, and at the end of sample run. Analysis of the IPC solution immediately following calibration must verify that the instrument is within ±10% of calibration. Subsequent analyses of the IPC solution must be within ±10% of calibration. If the calibration cannot be verified within the specified limits, reanalyze the IPC solution. If the second analysis of the IPC solution confirms calibration to be outside the limits, sample analysis must be discontinued, the cause determined, corrected and/or the instrument re-calibrated. All samples following the last acceptable IPC solution must be re-analyzed. The analysis data of the calibration blank and IPC solution must be kept on file with the sample analyses data.
14.0 Pollution Prevention

14.1 All analysts must follow the GeoLabs waste management plan in order to eliminate possibilities of pollution. All standards or samples that are known to contain any hazardous materials shall be disposed of as hazardous waste. See GeoLabs QAP for details on waste disposal.

15.0 Waste Management

15.1 Waste above regulatory levels shall be treated as hazardous waste. Follow GeoLabs waste disposal procedures as stated in GeoLabs QAP.

16.0 Detection Limits

16.1 Detection Limits must be determined as in CFR 136 App. B on a yearly basis. Current detection limits and reporting limits are kept on file with the Quality Assurance Manager.

17.0 Method Performance

17.1 Precision and accuracy data are available in the form of control charts updated on a monthly basis.

18.0 Data Assessment Criteria for Quality Control Measures

18.1 LCS values must be between the control chart limits or the method limit of 85-115%, whichever is tighter, or re-digestion and re-analysis of all samples in analytical batch must be performed after correction of problem.

18.2 The Instrument Performance Check solution (IPC) must be within 90-110% recovery initially and between 90-110% for subsequent analysis.

18.3 The Matrix Spike must be within 70-130%. Control charts must be used to assess precision between duplicate analysis. Problems with the Matrix Spike analysis shall be reported on the data report form.

19.0 Corrective Actions for Out-of-Control Data

19.1 If batch QC data is out of control the analyst must determine the cause of the problem, initiate correction, and re-analyze all data that is associated with the batch. For matrix problems associated with clients samples, report on the data report form.

20.0 Contingencies for Out-of-Control Data

20.1 If out-of-control data must be reported then the data must be qualified by the analyst. A note must be made on final report describing the situation.

21.0 References

Standard Test Method for Materials Finer than 75-μm (No. 200) Sieve in Mineral Aggregates by Washing

This standard is issued under the fixed designation C 117; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (e) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 This test method covers the determination of the amount of material finer than a 75-μm (No. 200) sieve in aggregate by washing. Clay particles and other aggregate particles that are dispersed by the wash water, as well as water-soluble materials, will be removed from the aggregate during the test.

1.2 Two procedures are included, one using only water for the washing operation, and the other including a wetting agent to assist the loosening of the material finer than the 75-μm (No. 200) sieve from the coarser material. Unless otherwise specified, Procedure A (water only) shall be used.

1.3 The values stated in SI units are to be regarded as the standard.

1.4 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:

C 136 Test Method for Sieve Analysis of Fine and Coarse Aggregates
C 670 Practice for Preparing Precision and Bias Statements for Test Methods for Construction Materials
C 702 Practice for Reducing Field Samples of Aggregate to Testing Size
D 75 Practice for Sampling Aggregates
E 11 Specification for Wire Cloth and Sieves for Testing Purposes

2.2 AASHTO Standard:

T11 Method of Test for Amount of Material Finer than 0.075-mm Sieve in Aggregate

3. Summary of Test Method

3.1 A sample of the aggregate is washed in a prescribed manner, using either plain water or water containing a wetting agent, as specified. The decanted wash water, containing suspended and dissolved material, is passed through a 75-μm (No. 200) sieve. The loss in mass resulting from the wash treatment is calculated as the percentage of the original sample and is reported as the percentage of material finer than a 75-μm (No. 200) sieve by washing.

4. Significance and Use

4.1 Material finer than the 75-μm (No. 200) sieve can be separated from larger particles much more efficiently and completely by wet sieving than through the use of dry sieving. Therefore, when accurate determinations of material finer than 75 μm in fine or coarse aggregate are desired, this test method is used on the sample prior to dry sieving in accordance with Test Method C 136. The results of this test method are included in the calculation in Test Method C 136, and the total amount of material finer than 75 μm by washing, plus that obtained by dry sieving the same sample, is reported with the results of Test Method C 136. Usually, the additional amount of material finer than 75 μm obtained in the dry sieving process is a small amount. If it is large, the efficiency of the washing operation should be checked. It could also be an indication of degradation of the aggregate.

4.2 Plain water is adequate to separate the material finer than 75 μm from the coarser material with most aggregates. In some cases, the finer material is adhering to the larger particles, such as some clay coatings and coatings on aggregates that have been extracted from bituminous mixtures. In these cases, the fine material will be separated more readily with a wetting agent in the water.

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1 Available from American Association of State Highway and Transportation Officials (AASHTO), 444 N. Capitol St., NW, Suite 249, Washington, DC 20001.

This test method is under the jurisdiction of ASTM Committee C19 on Concrete and Aggregates and is the direct responsibility of Subcommittee C19.10 on Normal Weight Aggregates.


2 For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For Annual Book of ASTM Standards volume information, refer to the standard's Documents Summary page on the ASTM website.
5. Apparatus and Materials

5.1 Balance—A balance or scale readable and accurate to 0.1 g or 0.1 % of the test load, whichever is greater, at any point within the range of use.

5.2 Sieves—A nest of two sieves, the lower being a 75-μm (No. 200) sieve and the upper a 1.18-mm (No. 16) sieve, both conforming to the requirements of Specification E 11.

5.3 Container—A pan or vessel of a size sufficient to contain the sample covered with water and to permit vigorous agitation without loss of any part of the sample or water.

5.4 Oven—An oven of sufficient size, capable of maintaining a uniform temperature of 110 ± 5 °C (230 ± 9 °F). 

5.5 Wetting Agent—Any dispersing agent, such as liquid dishwashing detergents, that will promote separation of the fine materials.

Note 1—The use of a mechanical apparatus to perform the washing operation is not precluded, provided the results are consistent with those obtained using manual operations. The use of some mechanical washing equipment with some samples may cause degradation of the sample.

6. Sampling

6.1 Sample the aggregate in accordance with Practice D 75. If the same test sample is to be tested for sieve analysis according to Test Method C 136, comply with the applicable requirements of that test method.

6.2 Thoroughly mix the sample of aggregate to be tested and reduce the quantity to an amount suitable for testing using the applicable methods described in Practice C 702. If the same test sample is to be tested according to Test Method C 136, the minimum mass shall be as described in the applicable sections of that method. Otherwise, the mass of the test sample, after drying, shall conform with the following:

<table>
<thead>
<tr>
<th>Nominal Maximum Size</th>
<th>Minimum Mass, g</th>
</tr>
</thead>
<tbody>
<tr>
<td>4.75 mm (No. 4) or smaller</td>
<td>300</td>
</tr>
<tr>
<td>Greater than 4.75 mm (No. 4) to 9.5 mm (½ in.)</td>
<td>1000</td>
</tr>
<tr>
<td>Greater than 9.5 mm (½ in.) to 19.0 mm (¾ in.)</td>
<td>2500</td>
</tr>
<tr>
<td>Greater than 19.0 mm (¾ in.)</td>
<td>5000</td>
</tr>
</tbody>
</table>

7. Selection of Procedure

7.1 Procedure A shall be used, unless otherwise specified by the specification with which the test results are to be compared, or when directed by the agency for which the work is performed.

8. Procedure A—Washing with Plain Water

8.1 Dry the test sample in the oven to constant mass at a temperature of 110 ± 5 °C (230 ± 9 °F). Determine the mass to the nearest 0.1 % of the mass of the test sample.

8.2 If the applicable specification requires that the amount passing the 75-μm (No. 200) sieve shall be determined on a portion of the sample passing a sieve smaller than the nominal maximum size of the aggregate, separate the sample on the designated sieve and determine the mass of the material passing the designated sieve to 0.1 % of the mass of this portion of the test sample. Use this mass as the original dry mass of the test sample in 10.1.

Note 2—Some specifications for aggregates with a nominal maximum size of 50 mm or greater, for example, provide a limit for material passing the 75-μm (No. 200) sieve determined on that portion of the sample passing the 25.6-mm sieve. Such procedures are necessary since it is impractical to wash samples of the size required when the same test sample is to be used for sieve analysis by Test Method C 136.

8.3 After drying and determining the mass, place the test sample in the container and add sufficient water to cover it. No detergent, dispersing agent, or other substance shall be added to the water. Agitate the sample with sufficient vigor to result in complete separation of all particles finer than the 75-μm (No. 200) sieve from the coarser particles, and to bring the fine material into suspension. Immediately pour the wash water containing the suspended and dissolved solids over the nested sieves, arranged with the coarsest sieve on top. Take care to avoid, as much as feasible, the decantation of coarser particles of the sample.

8.4 Add a second charge of water to the sample in the container, agitate, and decant as before. Repeat this operation until the wash water is clear.

Note 3—If mechanical washing equipment is used, the charging of water, agitating, and decanting may be a continuous operation.

8.5 Return all material retained on the nested sieves by flushing to the washed sample. Dry the washed aggregate in the oven to constant mass at a temperature of 110 ± 5 °C (230 ± 9 °F) and determine the mass to the nearest 0.1 % of the original mass of the sample.

Note 4—Following the washing of the sample and flushing any material retained on the 75-μm (No. 200) sieve back into the container, no water should be decanted from the container except through the 75-μm sieve, to avoid loss of material. Excess water from flushing should be evaporated from the sample in the drying process.

9. Procedure B—Washing Using a Wetting Agent

9.1 Prepare the sample in the same manner as for Procedure A.

9.2 After drying and determining the mass, place the test sample in the container. Add sufficient water to cover the sample, and add wetting agent to the water (Note 5). Agitate the sample with sufficient vigor to result in complete separation of all particles finer than the 75-μm (No. 200) sieve from the coarser particles, and to bring the fine material into suspension. Immediately pour the wash water containing the suspended and dissolved solids over the nested sieves, arranged with the coarsest sieve on top. Take care to avoid, as much as feasible, the decantation of coarser particles of the sample.

Note 5—There should be enough wetting agent to produce a small amount of foam when the sample is agitated. The quantity will depend on the hardness of the water and the quality of the detergent. Excessive suds may overflow the sieves and carry some material with them.

9.3 Add a second charge of water (without wetting agent) to the sample in the container, agitate, and decant as before. Repeat this operation until the wash water is clear.

9.4 Complete the test as for Procedure A.

10. Calculation

10.1 Calculate the amount of material passing a 75-μm (No. 200) sieve by washing as follows:

\[ A = \left( B - CYB \right) \times 100 \]  

(1)
where:
\[ A = \text{percentage of material finer than a 75-\mu m (No. 200) sieve by washing}, \]  
\[ B = \text{original dry mass of sample, g, and} \]  
\[ C = \text{dry mass of sample after washing, g}. \]

11. Report

11.1 Report the following information:
11.1.1 Report the percentage of material finer than the 75-\mu m (No. 200) sieve by washing to the nearest 0.1%, except if the result is 10% or more, report the percentage to the nearest whole number.
11.1.2 Include a statement as to which procedure was used.

12. Precision and Bias

12.1 Precision—The estimates of precision of this test method listed in Table 1 are based on results from the AASHTO Materials Reference Laboratory Proficiency Sample Program, with testing conducted by this test method and AASHTO Method T 11. The significant differences between the methods at the time the data were acquired is that Method T 11 required, while Test Method C 117 prohibited, the use of a wetting agent. The data are based on the analyses of more than 100 paired test results from 40 to 100 laboratories.

12.1.1 The precision values for fine aggregate in Table 1 are based on nominal 500-g test samples. Revision of this test method in 1994 permits the fine aggregate test sample size to be 300 g minimum. Analysis of results of testing of 300-g and 500-g test samples from Aggregate Proficiency Test Samples 99 and 100 (Samples 99 and 100 were essentially identical) produced the precision values in Table 2, which indicates only minor differences due to test sample size.

Note 6—The values for fine aggregate in Table 1 will be revised to reflect the 300-g test sample size when sufficient number of Aggregate Proficiency Tests have been conducted using that sample size to provide reliable data.

12.2 Bias—Since there is no acceptable reference material suitable for determining the bias for the procedure in this test method, no statement on bias is made.

13. Keywords

13.1 aggregate; coarse aggregate; fine aggregate; grading; loss by washing; 75 \mu m (No. 200) sieve; size analysis

<table>
<thead>
<tr>
<th>TABLE 1 Precision</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
</tr>
<tr>
<td>Standard Deviation (1σ), %</td>
</tr>
<tr>
<td>Coarse Aggregate</td>
</tr>
<tr>
<td>Simple-Operator Precision</td>
</tr>
<tr>
<td>Multilaboratory Precision</td>
</tr>
<tr>
<td>Fine Aggregate</td>
</tr>
<tr>
<td>Simple-Operator Precision</td>
</tr>
<tr>
<td>Multilaboratory Precision</td>
</tr>
</tbody>
</table>

4 These numbers represent the (1σ) and (2σ) limits as described in Practice C 637.
5 Precision estimates are based on aggregates having a nominal maximum size of 0.075 mm (% in.), with less than 1.5% finer than the 75-\mu m (No. 200) sieve.
6 Precision estimates are based on fine aggregates having 10 to 30% finer than the 75-\mu m (No. 200) sieve.

<table>
<thead>
<tr>
<th>TABLE 2 Precision Data for 300-g and 500-g Test Samples</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fine Aggregate</td>
</tr>
<tr>
<td>Sample Size</td>
</tr>
<tr>
<td>Test Result</td>
</tr>
<tr>
<td>300 g</td>
</tr>
</tbody>
</table>

AASHTO T11/ASTM C117
Total material passing the No. 200 sieve by washing (%)

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Standard Test Method for
Sieve Analysis of Fine and Coarse Aggregates

This standard is issued under the fixed designation C 136; the number immediately following the designation indicates the year of
original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval.
A superscript epsilon (e) indicates an editorial change since the last reapproval.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 This test method covers the determination of the particle size distribution of fine and coarse aggregates by sieving.

1.2 Some specifications for aggregates which reference this test method contain grading requirements including both coarse and fine fractions. Instructions are included for sieve analysis of such aggregates.

1.3 The values stated in SI units are to be regarded as the standard. The values in parentheses are provided for information purposes only. Specification E 11 designates the size of sieve frames with inch units as standard, but in this test method the frame size is designated in SI units exactly equivalent to the inch units.

1.4 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:

C 117 Test Method for Materials Finer than 75-μm (No. 200) Sieve in Mineral Aggregates by Washing
C 125 Terminology Relating to Concrete and Concrete Aggregates
C 637 Specification for Aggregates for Radiation-Shielding Concrete
C 670 Practice for Preparing Precision and Bias Statements for Test Methods for Construction Materials
C 702 Practice for Reducing Samples of Aggregate to Testing Size
D 703 Practice for Sampling Aggregates

2.2 AASHTO Standard:

AASHTO No. T 27 Sieve Analysis of Fine and Coarse Aggregates

3. Terminology

3.1 Definitions—For definitions of terms used in this standard, refer to Terminology C 125.

4. Summary of Test Method

4.1 A sample of dry aggregate of known mass is separated through a series of sieves of progressively smaller openings for determination of particle size distribution.

5. Significance and Use

5.1 This test method is used primarily to determine the grading of materials proposed for use as aggregates or being used as aggregates. The results are used to determine compliance of the particle size distribution with applicable specification requirements and to provide necessary data for control of the production of various aggregate products and mixtures containing aggregates. The data may also be useful in developing relationships concerning porosity and packing.

5.2 Accurate determination of material finer than the 75-μm (No. 200) sieve cannot be achieved by use of this test method alone. Test Method C 117 for material finer than 75-μm sieve by washing should be employed.

5.3 Refer to methods of sampling and testing in Specification C 637 for heavyweight aggregates.

6. Apparatus

6.1 Balances—Balances or scales used in testing fine and coarse aggregate shall have readability and accuracy as follows:

6.1.1 For fine aggregate, readable to 0.1 g and accurate to 0.1 g or 0.1% of the test load, whichever is greater, at any point within the range of use.

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* A Summary of Changes section appears at the end of this standard.

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6.1.2 For coarse aggregate, or mixtures of fine and coarse aggregate, readable and accurate to 0.5 g or 0.1% of the test load, whichever is greater, at any point within the range of use.

6.2 Sieves—The sieve cloth shall be mounted on substantial frames constructed in a manner that will prevent loss of material during sieving. The sieve cloth and standard sieve frames shall conform to the requirements of Specification E 11. Nonstandard sieve frames shall conform to the requirements of Specification E 11 as applicable.

Note 1—It is recommended that sieves mounted in frames larger than standard 203.2-mm (8 in.) diameter be used for testing coarse aggregate to reduce the possibility of overloading the sieves. See 6.3.

6.3 Mechanical Sieve Shaker—A mechanical sieving device, if used, shall create motion of the sieves to cause the particles to bounce, tumble, or otherwise turn so as to present different orientations to the sieving surface. The sieving action shall be such that the criterion for adequacy of sieving described in 8.4 is met in a reasonable time period.

Note 2—Use of a mechanical sieve shaker is recommended when the size of the sample is 20 kg or greater, and may be used for smaller samples, including fine aggregate. Excessive time (more than approximately 10 min) to achieve adequate sieving may result in degradation of the sample. The same mechanical sieve shaker may not be practical for all sizes of samples, since the large sieving area required for practical sieving of a large nominal size coarse aggregate very likely could result in loss of a portion of the sample if used for a small sample of coarse aggregate or fine aggregate.

6.4 Oven—An oven of appropriate size capable of maintaining a uniform temperature of 110 ± 5 °C (230 ± 9 °F).

7. Sampling

7.1 Sample the aggregate in accordance with Practice D 75. The size of the field sample shall be the quantity shown in Practice D 75 or four times the quantity required in 7.4 and 7.5 (except as modified in 7.6), whichever is greater.

7.2 Thoroughly mix the sample and reduce it to an amount suitable for testing using the applicable procedures described in Practice C 702. The sample for test shall be approximately the quantity desired when dry and shall be the end result of the reduction. Reduction to an exact predetermined quantity shall not be permitted.

Note 3—Where sieve analysis, including determination of material finer than the 75-μm sieve, is the only testing proposed, the size of the sample may be reduced in the field to avoid shipping excessive quantities of extra material to the laboratory.

7.3 Fine Aggregate—The size of the test sample, after drying, shall be 300 g minimum.

7.4 Coarse Aggregate—The size of the test sample of coarse aggregate shall conform with the following:

<table>
<thead>
<tr>
<th>Nominal Maximum Size,</th>
<th>Test Sample Size,</th>
</tr>
</thead>
<tbody>
<tr>
<td>Square Openings, mm (in)</td>
<td>min. kg (lb)</td>
</tr>
<tr>
<td>6.5 (1/4)</td>
<td>1 (2)</td>
</tr>
<tr>
<td>12.5 (1/2)</td>
<td>2 (4)</td>
</tr>
<tr>
<td>19.0 (3/4)</td>
<td>5 (11)</td>
</tr>
<tr>
<td>25.0 (1)</td>
<td>10 (22)</td>
</tr>
<tr>
<td>37.5 (1 1/2)</td>
<td>15 (33)</td>
</tr>
<tr>
<td>50 (2)</td>
<td>20 (44)</td>
</tr>
<tr>
<td>63 (2 1/2)</td>
<td>25 (55)</td>
</tr>
<tr>
<td>75 (3)</td>
<td>30 (66)</td>
</tr>
<tr>
<td>90 (3 1/4)</td>
<td>40 (88)</td>
</tr>
</tbody>
</table>

7.5 Coarse and Fine Aggregate Mixtures—The size of the test sample of coarse and fine aggregate mixtures shall be the same as for coarse aggregate in 7.4.

7.6 Samples of Large Size Coarse Aggregate—The size of sample required for aggregate with 50-mm nominal maximum size or larger is such as to preclude convenient sample reduction and testing as a unit except with large mechanical splitters and sieve shakers. As an option when such equipment is not available, instead of combining and mixing sample increments and then reducing the field sample to testing size, conduct the sieve analysis on a number of approximately equal sample increments such that the total mass tested conforms to the requirement of 7.4.

7.7 In the event that the amount of material finer than the 75-μm (No. 200) sieve is to be determined by Test Method C 117, proceed as follows:

7.7.1 For aggregates with a nominal maximum size of 12.5 mm (1/2 in.) or less, use the same test sample for testing by Test Method C 117 and this test method. First test the sample in accordance with Test Method C 117 through the final drying operation, then dry sieve the sample as stipulated in 8.2.8.7 of this test method.

7.7.2 For aggregates with a nominal maximum size greater than 12.5 mm (1/2 in.), use a single test sample as described in 7.7.1, or optionally use separate test samples for Test Method C 117 and this test method.

7.7.3 Where the specifications require determination of the total amount of material finer than the 75-μm sieve by washing and dry sieving, use the procedure described in 7.7.1.

8. Procedure

8.1 Dry the sample to constant mass at a temperature of 110 ± 5 °C (230 ± 9 °F).

Note 4—For control purposes, particularly where rapid results are desired, it is generally not necessary to dry coarse aggregate for the sieve analysis test. The results are little affected by the moisture content unless:

(1) the nominal maximum size is smaller than about 12.5 mm (1/2 in.): (2) the coarse aggregate contains appreciable material finer than 4.75 mm (No. 4); or (3) the coarse aggregate is highly absorptive (a lightweight aggregate, for example). Also, samples may be dried at the higher temperatures associated with the use of hot plates without affecting results, provided steam escapes without generating pressures sufficient to fracture the particles, and temperatures are not so great as to cause chemical breakdown of the aggregate.

8.2 Select sieves with suitable openings to furnish the information required by the specifications covering the material to be tested. Use additional sieves as desired or necessary to provide other information, such as fineness modulus, or to regulate the amount of material on a sieve. Nest the sieves in order of decreasing size of opening from top to bottom and place the sample on the top sieve. Agitate the sieves by hand or by mechanical apparatus for a sufficient period, established by trial or checked by measurement on the actual test sample, to meet the criterion for adequacy or sieving described in 8.4.

8.3 Limit the quantity of material on a given sieve so that all particles have opportunity to reach sieve openings a number of times during the sieving operation. For sieves with openings
smaller than 4.75-mm (No. 4), the quantity retained on any sieve at the completion of the sieving operation shall not exceed 7 kg/m² of sieving surface area (Note 5). For sieves with openings 4.75 mm (No. 4) and larger, the quantity retained in kg shall not exceed the product of 2.5 × (sieve opening, mm × effective sieving area, m²). This quantity is shown in Table 1 for five sieve-frame dimensions in common use. In no case shall the quantity retained be so great as to cause permanent deformation of the sieve cloth.

8.3.1 Prevent an overload of material on an individual sieve by one of the following methods:

8.3.1.1 Insert an additional sieve with opening size intermediate between the sieve that may be overloaded and the sieve immediately above that sieve in the original set of sieves.

8.3.1.2 Split the sample into two or more portions, sieving each portion individually. Combine the masses of the several portions retained on a specific sieve before calculating the percentage of the sample on the sieve.

8.3.1.3 Use sieves having a larger frame size and providing greater sieving area.

Note 5—The 7 kg/m² amounts to 200 g for the usual 203.2-mm (8-in.) diameter sieve (with effective sieving surface diameter of 190.5 mm (7.5 in.).

8.4 Continue sieving for a sufficient period and in such manner that, after completion, not more than 1% by mass of the material retained on any individual sieve will pass that sieve during 1 min of continuous hand sieving performed as follows: Hold the individual sieve, provided with a snug-fitting pan and cover, in a slightly inclined position in one hand. Strike the side of the sieve sharply and with an upward motion against the heel of the other hand at the rate of about 150 times per minute, run the sieve about one sixth of a revolution at

intervals of about 25 strokes. In determining sufficiency of sieving for sizes larger than the 4.75-mm (No. 4) sieve, limit the material on the sieve to a single layer of particles. If the size of the mounted testing sieves makes the described sieving motion impractical, use 203-mm (8 in.) diameter sieves to verify the sufficiency of sieving.

8.5 In the case of coarse and fine aggregate mixtures, refer to 8.3.1 to prevent overloading of individual sieves.

8.5.1 Optionally, reduce the portion finer than the 4.75-mm (No. 4) sieve using a mechanical splitter according to Practice C 702. If this procedure is followed, compute the mass of each size increment of the original sample as follows:

\[
A = \frac{W}{W_t} \times B
\]

where:

- \( A \) = mass of size increment on total sample basis,
- \( W_t \) = mass of fraction finer than 4.75-mm (No. 4) sieve in total sample,
- \( W_s \) = mass of reduced portion of material finer than 4.75-mm (No. 4) sieve actually sieved, and
- \( B \) = mass of size increment in reduced portion sieved.

8.6 Unless a mechanical sieve shaker is used, hand sieve particles larger than 75 mm (3 in.) by determining the smallest sieve opening through which each particle will pass. Start the test on the smallest sieve to be used. Rotate the particles, if necessary, in order to determine whether they will pass through a particular opening; however, do not force particles to pass through an opening.

8.7 Determine the mass of each size increment on a scale or balance conforming to the requirements specified in §1.1 to the nearest 0.1% of the total original dry sample mass. The total mass of the material after sieving should check closely with original mass of sample placed on the sieves. If the amounts differ by more than 0.3%, based on the original dry sample mass, the results should not be used for acceptance purposes.

8.8 If the sample has previously been tested by Test Method C 117, add the mass finer than the 75-μm (No. 200) sieve determined by that test method to the mass passing the 75-μm (No. 200) sieve by dry sieving of the same sample in this test method.

9. Calculation

9.1 Calculate percentages passing, total percentages retained, or percentages in various size fractions to the nearest 0.1% on the basis of the total mass of the initial dry sample. If the same test sample was first tested by Test Method C 117, include the mass of material finer than the 75-μm (No. 200) size by washing in the sieve analysis calculation; and use the total dry sample mass prior to washing in Test Method C 117 as the basis for calculating all the percentages.

9.1.1 When sample increments are tested as provided in 7.6, total the masses of the portion of the increments retained on each sieve, and use these masses to calculate the percentages as in 9.1.

9.2 Calculate the fineness modulus, when required, by adding the total percentages of material in the sample that is coarser than each of the following sieves (cumulative percentages retained), and dividing the sum by 100: 150-μm (No.
100), 300-μm (No. 50), 600-μm (No. 30), 1.18-mm (No. 16), 2.36-mm (No. 8), 4.75-mm (No. 4), 9.5-mm (3/8-in.), 19.0-mm (5/8-in.), 37.5-mm (1 1/2-in.), and larger increasing in the ratio of 2 to 1.

10. Report

10.1 Depending upon the form of the specifications for use of the material under test, the report shall include the following:

10.1.1 Total percentage of material passing each sieve, or

10.1.2 Total percentage of material retained on each sieve, or

10.1.3 Percentage of material retained between consecutive sieves.

10.2 Report percentiles to the nearest whole number, except if the percentage passing the 75-μm (No. 200) sieve is less than 10%, it shall be reported to the nearest 0.1%.

10.3 Report the fineness modulus, when required, to the nearest 0.01.

11. Precision and Bias

11.1 Precision—The estimates of precision for this test method are listed in Table 2. The estimates are based on the results from the AASHTO Materials Reference Laboratory Proficiency Sample Program, with testing conducted by Test Method C 136 and AASHTO No. T 77. The data are based on the analyses of the test results from 65 to 233 laboratories that tested 18 pairs of coarse aggregate proficiency test samples and test results from 74 to 222 laboratories that tested 17 pairs of fine aggregate proficiency test samples (Samples No. 21 through 90). The values in the table are given for different ranges of total percentage of aggregate passing a sieve.

11.1.1 The precision values for fine aggregate in Table 2 are based on nominal 500-g test samples. Revision of this test method in 1994 permits the fine aggregate test sample size to be 500 g minimum. Analysis of results of testing of 300-g and 500-g test samples from Aggregate Proficiency Test Samples 99 and 100 (Samples 99 and 100 were essentially identical) produced the precision values in Table 2, which indicate only minor differences due to test sample size.

Note 6—The values for fine aggregate in Table 2 will be revised to reflect the 300-g test sample size when a sufficient number of Aggregate Proficiency Tests have been conducted using that sample size to provide reliable data.

TABLE 2 Precision

<table>
<thead>
<tr>
<th>Coarse Aggregate, a</th>
<th>Total Percentage of Material Passing</th>
<th>Standard Deviation (1σ), % a</th>
<th>Acceptable Range of Two Results (2γ), % a</th>
</tr>
</thead>
<tbody>
<tr>
<td>Single-operator precision</td>
<td>&lt;100 ±85</td>
<td>0.32</td>
<td>0.9</td>
</tr>
<tr>
<td></td>
<td>&lt;95 ±85</td>
<td>0.81</td>
<td>2.3</td>
</tr>
<tr>
<td></td>
<td>&lt;85 ±85</td>
<td>1.14</td>
<td>3.8</td>
</tr>
<tr>
<td></td>
<td>&lt;75 ±80</td>
<td>2.09</td>
<td>6.4</td>
</tr>
<tr>
<td></td>
<td>&lt;65 ±80</td>
<td>1.32</td>
<td>3.7</td>
</tr>
<tr>
<td></td>
<td>&lt;55 ±65</td>
<td>0.96</td>
<td>2.7</td>
</tr>
<tr>
<td></td>
<td>&lt;45 ±60</td>
<td>1.09</td>
<td>2.8</td>
</tr>
<tr>
<td></td>
<td>&lt;35 ±55</td>
<td>0.75</td>
<td>2.1</td>
</tr>
<tr>
<td></td>
<td>&lt;25 ±50</td>
<td>0.53</td>
<td>1.5</td>
</tr>
<tr>
<td></td>
<td>&lt;15 ±50</td>
<td>0.27</td>
<td>0.8</td>
</tr>
<tr>
<td></td>
<td>&lt;10 ±50</td>
<td>0.35</td>
<td>1.0</td>
</tr>
</tbody>
</table>

Multi-laboratory precision

<table>
<thead>
<tr>
<th>Fine Aggregate, a</th>
<th>Total Percentage of Material Passing</th>
<th>Standard Deviation (1σ), % a</th>
<th>Acceptable Range of Two Results (2γ), % a</th>
</tr>
</thead>
<tbody>
<tr>
<td>Single-operator precision</td>
<td>&lt;100 ±85</td>
<td>0.26</td>
<td>0.7</td>
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<td>&lt;95 ±85</td>
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<td>1.8</td>
</tr>
<tr>
<td></td>
<td>&lt;1 ±50</td>
<td>0.31</td>
<td>0.9</td>
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a These numbers represent, respectively, the (1σ) and (2σ) limits described in Practice C 070.

b The precision estimates are based on aggregates with nominal maximum size of 15.0 mm (5/8 in.).

11.2 Bias—Since there is no accepted reference material suitable for determining the bias in this test method, no statement on bias is made.

12. Keywords

12.1 aggregate; coarse aggregate; fine aggregate; grading; grading; sieve analysis; size analysis
<table>
<thead>
<tr>
<th>Test Method C 136/AASHTO No. 127</th>
<th>Sample Size</th>
<th>Number Labs</th>
<th>Average</th>
<th>1s</th>
<th>d/ds</th>
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<th>d/ds</th>
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<td>Total material passing the No. 4 sieve (%)</td>
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<td>300 g</td>
<td>276</td>
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<td>300 g</td>
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**SUMMARY OF CHANGES**

Committee C09 has identified the location of selected changes to this test method since the last issue, C 136-05, that may impact the use of this test method. (Approved February 15, 2006)

(1) Revised 8.5.1.
(2) Revised 7.7.1.
(3) Revised 8.5.

Committee C09 has identified the location of selected changes to this test method since the last issue, C 136-04, that may impact the use of this test method. (Approved February 1, 2005)

(1) Added 5.3.
(2) Revised 8.5.

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METHOD 9060A

TOTAL ORGANIC CARBON

1.0 SCOPE AND APPLICATION

1.1 This method is used to determine the concentration of organic carbon in ground water, surface and saline waters, and domestic and industrial wastes. Some restrictions are noted in Secs. 2.0 and 3.0.

1.2 This method is most applicable to measurement of organic carbon above 1 mg/L.

2.0 SUMMARY OF METHOD

2.1 Organic carbon is measured using a carbonaceous analyzer. This instrument converts the organic carbon in a sample to carbon dioxide (CO₂) by either catalytic combustion or wet chemical oxidation. The CO₂ formed is then either measured directly by an infrared detector or converted to methane (CH₄) and measured by a flame ionization detector. The amount of CO₂ or CH₄ in a sample is directly proportional to the concentration of carbonaceous material in the sample.

2.2 Carbonaceous analyzers are capable of measuring all forms of carbon in a sample. However, because of various properties of carbon-containing compounds in liquid samples, the manner of preliminary sample treatment as well as the instrument settings will determine which forms of carbon are actually measured. The forms of carbon that can be measured by this method are:

1. Soluble, nonvolatile organic carbon: e.g., natural sugars.

2. Soluble, volatile organic carbon: e.g., mercaptans, alkanes, low molecular weight alcohols.

3. Insoluble, partially volatile carbon: e.g., low molecular weight oils.

4. Insoluble, particulate carbonaceous materials: e.g., cellulose fibers.

5. Soluble or insoluble carbonaceous materials adsorbed or entrapped on insoluble inorganic suspended matter: e.g., oily matter adsorbed on soil particles.

2.3 Carbonate and bicarbonate are inorganic forms of carbon and must be separated from the total organic carbon value. Depending on the instrument manufacturer's instructions, this separation can be accomplished by either a simple mathematical subtraction, or by removing the carbonate and bicarbonate by converting them to CO₂ with degassing prior to analysis.

3.0 INTERFERENCES

3.1 Carbonate and bicarbonate carbon represent an interference under the terms of this test and must be removed or accounted for in the final calculation.
3.2 This procedure is applicable only to homogeneous samples which can be injected into the apparatus reproducibly by means of a microtiter-type syringe or pipet. The openings of the syringe or pipet limit the maximum size of particle which may be included in the sample.

3.3 Removal of carbonate and bicarbonate by acidification and purging with nitrogen, or other inert gas, can result in the loss of volatile organic substances.

4.0 APPARATUS AND MATERIALS

4.1 Apparatus for blending or homogenizing samples -- Generally, a Waring-type blender is satisfactory.

4.2 Apparatus for total and dissolved organic carbon

4.2.1 Several companies manufacture analyzers for measuring carbonaceous material in liquid samples. The most appropriate system should be selected based on consideration of the types of samples to be analyzed, the expected concentration range, and the forms of carbon to be measured.

4.2.2 No specific analyzer is recommended as superior. If the technique of chemical oxidation is used, the laboratory must be certain that the instrument is capable of achieving good carbon recoveries in samples containing particulates.

5.0 REAGENTS

5.1 ASTM Type II water (ASTM D1193) -- Water should be monitored for impurities, and should be boiled and cooled to remove CO₂.

5.2 Potassium hydrogen phthalate, stock solution, 1,000 mg/L carbon -- Dissolve 0.2128 g of potassium hydrogen phthalate (primary standard grade) in Type II water and dilute to 100.0 mL.

NOTE: Sodium oxalate and acetic acid are not recommended as stock solutions.

5.3 Potassium hydrogen phthalate, standard solutions -- Prepare standard solutions from the stock solution by dilution with Type II water.

5.4 Carbonate-bicarbonate, stock solution, 1,000 mg/L carbon -- Weigh 0.3500 g of sodium bicarbonate and 0.4418 g of sodium carbonate and transfer both to the same 100-mL volumetric flask. Dissolve with Type II water.

5.5 Carbonate-bicarbonate, standard solution -- Prepare a series of standards similar to Step 5.3.

NOTE: This standard is not required by some instruments.

5.6 Blank solution -- Use the same Type II water as was used to prepare the standard solutions.
6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 Sampling and storage of samples in glass bottles is preferable. Sampling and storage in plastic bottles such as conventional polyethylene and cubitainers is permissible if it is established that the containers do not contribute contaminating organics to the samples.

NOTE: A brief study performed in the EPA Laboratory indicated that Type II water stored in new, 1-qt cubitainers did not show any increase in organic carbon after 2 weeks' exposure.

6.2 Because of the possibility of oxidation or bacterial decomposition of some components of aqueous samples, the time between sample collection and the start of analysis should be minimized. Also, samples should be kept cool (4 • C) and protected from sunlight and atmospheric oxygen.

6.3 In instances where analysis cannot be performed within 2 hr from time of sampling, the sample is acidified (pH • 2) with HCl or H₂SO₄.

7.0 PROCEDURE

7.1 Homogenize the sample in a blender.

NOTE: To avoid erroneously high results, inorganic carbon must be accounted for. The preferred method is to measure total carbon and inorganic carbon and to obtain the organic carbon by subtraction. If this is not possible, follow Steps 7.2 and 7.3 prior to analysis; however, volatile organic carbon may be lost.

7.2 Lower the pH of the sample to 2.

7.3 Purge the sample with nitrogen for 10 min.

7.4 Follow instrument manufacturer's instructions for calibration, procedure, and calculations.

7.5 For calibration of the instrument, a series of standards should be used that encompasses the expected concentration range of the samples.

7.6 Quadruplicate analysis is required. Report both the average and the range.

8.0 QUALITY CONTROL

8.1 All quality control data should be maintained and available for easy reference or inspection.

8.2 Employ a minimum of one blank per sample batch to determine if contamination or any memory effects are occurring.

8.3 Verify calibration with an independently prepared check standard every 15 samples.

8.4 Run one spike duplicate sample for every 10 samples. A duplicate sample is a sample brought through the whole sample preparation and analytical process.
9.0  METHOD PERFORMANCE

9.1  Precision and accuracy data are available in Method 415.1 of Methods for Chemical
Analysis of Water and Wastes.

10.0 REFERENCES


2.  Standard Methods for the Examination of Water and Wastewater, 14th ed., p. 532,
Method 505 (1975).
Method 9060A
TOTAL ORGANIC CARBON

Start

7.1 Homogenize the sample in a blender.

7.2 Lower the sample pH.

7.3 Purge the sample with nitrogen.

A

7.4 Follow manufacturer's instructions for calibration procedure, and calculations using carbonaceous analyzer.

7.5 Use series of standards for calibration.

7.6 Quadruplicate analysis.

Stop
GEOLABS, Inc.
Standard Operating Procedure

Determination of Mercury in Solid or Semisolid Waste
Revision: 3.1

EPA Method 7471A

Effective Date 8/25/2006

Approved by:

Mark Perkins; Quality Assurance Manager

Jim Chen; Laboratory Director
STANDARD OPERATING PROCEDURE

Determination of Mercury in Solid or Semi-solid Waste by Cold Vapor Atomic Absorption Spectrometry

1.0 Scope and Application

1.1 This procedure measures total mercury in soils, sediments, and sludges.

2.0 Summary of Method

2.1 Prior to analysis, the solid or semi-solid samples must be prepared according to the procedures discussed in this method.

2.2 With the use of a Leeman Labs PS200i Mercury Analyzer, the mercury is reduced to the elemental state and aedated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of an atomic absorption spectrophotometer. Absorbance (peak height) is measured as a function of mercury concentration.

3.0 Definitions

3.1 Calibration Blank: A volume of reagent water acidified with the same acid matrix as in the calibration standards. The calibration blank is a zero standard.

3.2 Method Blank: A volume of reagent water that is digested exactly as a sample. The method blank is used to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus.

3.3 Laboratory Control Sample: A volume of reagent water to which known amounts of method analytes are added. The LCS is analyzed exactly like a sample, and its purpose is to determine the laboratory accuracy for the method.

3.4 Matrix Spike Sample: A volume of an environmental sample to which known amounts of method analytes are added. The MS is analyzed exactly like a sample and its purpose is to determine whether the sample matrix contributes bias to the analytical results. The MS is normally analyzed in duplicate (MS/MSD).

3.5 Calibration Standard: A solution prepared from the dilution of stock standard solutions. The CAL solns. Are used to calibrate the instrument.

3.6 Cal. Check: A solution with a known amount of analyte, which is run after every 10 samples to check the calibration of the instrument.

3.7 Working Standard: A 100-ppb standard from which the CAL STD's are diluted.

4.0 Interferences

4.1 Interferences have been reported for waters containing sulfide, chloride, copper and tellurium, Organic compounds with broad band UV absorbance are also interferences.
5.0 Safety

5.1 See GeoLabs Chemical Hygiene Plan for information on laboratory safety.

6.0 Apparatus and Materials

6.1 Leeman labs PS200 II Mercury analyzer

6.2 Water Bath - covered, and capable of maintaining 95°C.

6.3 BOD Bottles

6.4 Glassware: Class A volumetric flasks and graduated cylinders

6.5 Assorted Class A Volumetric pipets.

6.6 Analytical Balance

7.0 Reagents and Standards

7.1 De-ionized water (DI)

7.2 Nitric Acid (concentrated), HNO3

7.3 Hydrochloric Acid (concentrated), HCl

7.4 Aqua Regia: (3:1) Add 3.75mL of conc. HCL plus 1.25 mL of conc. HNO3

7.5 1000 mg/L Hg standard

7.4.1 100 ppb Cal Std (1.0 ml into 100 ml 5% HNO3)

7.6 Potassium permanganate, 5% (w/v) (Dissolve 50 grams into 1L DI water)

7.7 Sodium chloride hydroxylamine sulfate (Dissolve 120 g NaCl and 120 g hydroxylamine sulfate into 1000 ml DI water)

7.8 Mercury stock solution (10ppm): Take 1mL of 1000ppm into 100mL of 5% HNO3

7.9 Mercury working standard (1000ppb): Take 1mL of the 10ppm stock solution into 100mL of 5% HNO3, this must be prepared daily

7.10 Stannous chloride, 10% (w/v) (Dissolve 50 g into 500mL 15% H2SO4)

8.0 Sample Collection, Preservation, and Handling

8.1 Samples should be collected in a glass jar with a Teflon liner. Non-aqueous samples shall be refrigerated and analyzed as soon as possible. Samples must be analyzed within 28 days of collection.
9.0 Procedure

9.1 Sample Preparation: Weigh out 0.2g portions of well-mixed homogeneous sample and place them into BOD bottles. Add 5 ml of reagent water and 5ml of aqua regia (3.75 ml of HCl and 1.25 ml of HNO3). Heat 2 minutes in a water bath at 95°C. Cool and add 50 ml reagent water and 15 ml potassium permanganate solution to each sample bottle. Mix thoroughly and place in the water bath for 30 minutes at 95°C. Cool and add 6 ml of sodium chloride-hydroxylamine sulfate to reduce the excess permanganate. Do this addition under a hood as C12 could be evolved. Add 55ml of reagent water. The sample is now ready for analysis by Leeman PS20011 Automated Mercury Analyzer.

9.2 Standard Preparation: Transfer 0.0, 0.5, 1.0, 2.0, 5.0, and 10ml aliquots of the mercury working standard, containing 0-1.0 mg of mercury, to a series of BOD bottles. Add enough reagent water to each bottle to make a total volume of 10 ml. Add 5 ml of aqua regia, 50 ml DI water, 15 ml of KMNO4 solution and 6 ml of sodium chloride-hydroxylamine sulfate solution to reduce the excess permanganate. Add 55 ml of DI water. The standard is now ready for analysis by Leeman PS20011 Automated Mercury Analyzer.

9.3 Analysis: Turn on the PS20011. Turn on the Hg lamp and allow to warm-up for 20 minutes. Open the Run Protocol and a Work Folder. Type in the sequence of samples for the run. Run the curve, if the correlation coefficient is 0.995 or better, accept the curve and print the screen. Run the samples and QC.

Note: For all procedural details related to the PS20011 operation see Appendix 1:PS20011 Automated Mercury Analyzer Operations Sub Manual.

9.4 Calculation: Divide the results by (weight (g) X %solid). Report results in mg/Kg.

10. Quality Control

10.1 Each analyst using this method is required to follow the formal quality control program as described in this SOP and the current version of the GEOLABS QA PLAN. The requirements of this program include the demonstration of capability, and the periodic analysis of method blanks and laboratory control samples (LCS). The analyst is required to maintain and use performance records in the form of control charts, to define the quality of the data that is generated, for the purpose of acceptance.

10.2 Demonstration of Capability- Prior to acceptance and institution of any test method, satisfactory demonstration of method capability is required. A demonstration of capability must be completed each time there is a significant change in instrument type, personnel, or test method. See the current version of the GEOLABS QA PLAN for details on completing a demonstration of capability.

10.3 Method Blanks (MB)- The analyst must analyze at least one Method Blank with each batch of samples per matrix per sample preparation method. The results of this analysis shall be one of the QC measures to be used to assess batch acceptance. The source of the contamination must be investigated and measures taken to correct, minimize or eliminate the problem if (1) the blank contamination exceeds a concentration greater than 1/10 of the measured concentration of any sample in the associated sample batch or (2) the blank contamination exceeds the concentration present in the samples and is greater than 1/10 of the specified regulatory limit. Any sample associated with the contaminated blank shall be reprocessed for analysis or the results reported with appropriate data qualifying codes.
10.4 Laboratory Control Sample (LCS). The LCS shall be analyzed at a minimum of 1 per batch of 10 or less samples per matrix type per sample preparation method. The results of these samples shall be used to determine batch acceptance by comparison to control chart limits for percent recovery. The LCS must fall within the 85-115% range. See GEOLABS QA PLAN for control chart performance data preparation.

10.5 Matrix Spike (MS). Shall be performed at a frequency of one in 10 samples per matrix type per sample preparation method. The selected sample shall be rotated among client samples so that various matrix problems may be noted and/or addressed. Poor performance in a matrix spike may indicate a problem with the sample composition and shall be reported to the client whose sample was used for the spike.

10.6 Matrix Spike Duplicate (MSD) or Sample Duplicates. Shall be analyzed at a minimum of 1 in 10 samples per sample preparation method. The selected sample shall be rotated among clients so that various matrix problems may be noted and/or addressed. Poor performance in the duplicates may indicate a problem with the sample composition and shall be reported to the client whose sample was used for the duplicate. Duplicate results should be compared to control chart limits for replicate analysis to determine acceptable reproducibility.

11.0 Pollution Prevention

11.1 All analysts are urged to follow the GeoLabs waste management plan in order to eliminate possibilities of pollution. All standards or samples tested positive for mercury above regulatory levels shall be disposed of as hazardous waste. See GeoLabs QAP for details on waste disposal.

12.0 Waste Management

12.1 Waste mercury above regulatory levels shall be treated as hazardous waste. Follow GeoLabs waste disposal procedures as stated in GeoLabs QAP.

13.0 Detection Limit

13.1 Detection Limits must be determined as in CFR 136 App. B, on a yearly basis. The reporting limit for mercury has been set at 0.02 mg/Kg.

14.0 Method Performance

14.1 Precision and accuracy data are available in the form of control charts updated on a monthly basis.

15.0 Data Assessment criteria for quality control measures

15.1 A calibration reference standard run immediately after the curve must be within 10% of its true value.

15.2 After every 10 samples a mid-range standard must be within 20% of its true value.

15.3 Do not report data below the determined analyte MDL.

15.4 Report analyte concentrations up to three significant figures.

15.5 All blanks must be < the MDL.
16.0 Corrective Actions for Out-of-Control Data

16.1 If any data is out of control the analyst must determine the cause of the problem, initiate correction, and re-analyze all data that is associated with the batch.

17.0 Contingencies for out-of-control data

17.1 If out-of-control data must be reported then the data must be qualified by the analyst. A note must be made on final report describing the situation.

18.0 References

STANDARD OPERATING PROCEDURE
Extractable Petroleum Hydrocarbons
MADEP EPH

1.0 Scope and Application

This Standard Operating Procedure (SOP) is designed to measure the collective concentrations of extractable aliphatic and aromatic petroleum hydrocarbons in water and soil matrices.

2.0 Summary of Method

Water samples are extracted with Dichloromethane (DCM) using a separatory funnel. The extract is fractionated into aliphatic and aromatic portions and analyzed using a GC/ FID. The aromatics fraction is also analyzed for target PAH compounds using a GC/MSD.

Soil samples are extracted with Dichloromethane (DCM) using soxhlet extraction setup.

3.0 Preservation and Holding Times

Samples and sample extracts are stored at 4 C +/- 2 C in a contaminant free environment. Samples and standards are stored separately. If the sample will not be extracted within 24 hours, then adjust the pH to < 2.0 with H2SO4.

Extraction of the sample must be performed within 14 days of sampling for water and 7 days for soils. Extracts must be analyzed within 40 days.

4.0 Interference

Method interference may be caused by contaminants in sampling equipment, solvents, glassware, and Sep-Paks. Routinely, all of these things are demonstrated to be free from interference under the conditions of the analysis by the running of method blanks. Matrix interference may be caused by contaminants that are coextracted from the sample. Glassware contamination can be avoided by washing with hot soapy water, rinsing with DI water, and then rinsing all glassware used in the extraction with DCM before beginning extraction. Severe contamination is found in the Sep-Paks so they must be rinsed
with both extraction solvents (mainly for commercial Sep-Paks).

5.0 Definitions

5.1 Lab Blank - An analytical control sample prepared from an analyte free matrix (reagent water) to which all reagents are added in the same volume or proportions as used in sample processing, that is carried through the entire analytical procedure. The lab blank is used to document the level of laboratory background and reagent contamination resulting from the analytical process.

5.2 LCS - Laboratory Control Spike sample is an aliquot of organic free reagent water fortified with the spiking solution.

5.3 Duplicate - Sample duplicate is separate from the sample itself, although the two were taken from the same source. They must be from separate collection bottles. If no duplicate is available then the chemist must extract two LCS samples. This gives a measure of the precision associated with the lab procedures.

5.4 Matrix Spike Samples - Organic compounds of known quantities spiked into sample(s) in order to determine if the sample matrix contributes bias to the analytical results.

6.0 Apparatus and Materials

6.1 Separatory Funnel-2000ml with Teflon stopcock

6.2 Erlenmeyer Flasks-250ml

6.3 500ml round bottom flasks

6.4 Aluminum weighing dishes

6.5 Glass Funnels

6.6 Graduated Cylinder-1000ml

6.7 pH paper-indicator strips, pH 0-12

6.8 Glass Wool-silane treated

6.9 Soxhlet extraction setup
6.10 500ml concentrator reservoirs
6.11 Kuderna-Danish tube (KD tubes)-25ml
6.12 Snyder columns-three ball
6.13 Snap Clamps
6.14 Syringe-1.0ml
6.15 40ml vials with teflon-lined cap

7.0 Reagents and Standards

7.1 Dichloromethane (DCM)
7.2 Hexane
7.3 Acetone
7.4 H2SO4-1:1
7.5 Sodium Sulfate (Na2SO4)-granular anhydrous reagent grade, baked for 4 hours at 400 C, cooled, and stored in a glass jar.

7.6 Standards-Standard solutions may be prepared from pure materials or purchased as certified solutions. Standards are prepared in Acetone, Hexane, or DCM and stored at 4 C. Standards should be checked frequently for stability, and be replaced when comparison with second source standards indicate a problem or within six months.

7.6.1 Spiking Solution - Purchased from Ultra Scientific at 50ug/ml containing aliphatic and aromatic compounds. 1.0 ml of the spiking solution is added to the LCS and the matrix spike sample. The Spike concentration is 50ug/ml.

7.6.2 Surrogate Solution-Purchased from Ultra Scientific at 40ug/ml containing COD and OTP. 1.0 ml of the surrogate solution is added to all samples. The surrogate solution concentration is 40ug/ml.

7.6.3 Fractionation Surrogate-Purchased from Ultra
Scientific at 40μg/ml. The fractionation surrogate contains 2-Fluorobiphenyl and 2,2'-Difluorobiphenyl. 0.5 ml of the fractionation surrogate is added to each sample. The fractionation surrogate concentration is 40μg/ml.

8.0 Procedures

8.1 Liquid Extraction

8.1.1 Review project information by checking the chain of custody. Record sample numbers and information in Organic Preparation Logbook.

8.1.2 Clean hood work area to avoid cross contamination.

8.1.3 Rinse all glassware 3X with Acetone and 3X with DCM.

8.1.4 Place a small amount of glass wool inside the rinsed funnel and fill halfway with sodium sulfate. Rinse down the sodium sulfate with DCM. Place on top of the rinsed Erlenmeyer.

8.1.5 Retrieve samples from the organic Sample Storage Refrigerator.

8.1.6 Compare sample ID numbers with the Chain of Custody.

8.1.7 Label Separatory funnels and Erlenmeyer flasks with corresponding sample ID numbers using a black sharpie.

8.1.8 Pour entire sample jar into Separatory funnel, measuring exactly 1000 ml of reagent water into the method blank and LCS.

8.1.9 Create a duplicate by choosing one sample (preferably one that appears to be ND) and splitting it in half, extract and analyze both halves identically.

8.1.10 Measure the pH of the samples and record it in the Organic Preparations Logbook.

8.1.11 Add 1.0ml of surrogate to all samples of the analytical batch. Add 1.0ml of spike to all samples of the analytical batch selected for matrix spiking.

8.1.12 Measure 60 ml of DCM into each separatory funnel. Cap and
carefully invert while holding cap tightly. Vent into hood by
opening stopcock and swirling sample a few times. Close stop
cock and shake a few times. Vent the sample. Repeat until no gas
escapes when stop cock is opened. Shake for two minutes.

8.1.13 After two minutes, replace separatory funnels on the rack, open
tops, and allow to settle for a minimum of 5 min.

8.1.14 After all separatory funnels have been shaken for two minutes,
 emissions, drain the bottom layer into the corresponding glass funnel.

8.1.15 Repeat 8.1.12-8.1.14 two more times, only shaking for 1 minute,
for a total of three shakes (2min., 1min., 1min.).

8.1.16 After all samples have been shaken and drained 3X, rinse the
funnels with about 5-10 ml DCM.

8.1.17 Cap extract and store in refrigerator until ready to concentrate.

8.1.18 Pour sample volume into 1000 ml graduated cylinder and record
measurement in Organic Preparation Logbook.

8.2 *Soil Extraction by Soxhlet*

8.2.1 Weigh out 10.0g of sample in a small aluminum weighing dish and
dry sample with a small amount of Na2SO4. Place in extraction
thimble.

8.2.2 Using a syringe add 1.0ml of surrogate to all samples of the
analytical batch. Add 1.0ml of spike to all samples of the
analytical batch selected for matrix spiking.

8.2.3 Place 300mls of DCM into a 500ml round bottom flask containing
1 or 2 teflon boiling chips. Attach flask to soxlet extraction setup
and extract for 4hrs.

8.2.4 Allow sample to cool and remove from soxlet extraction setup.

8.2.5 Samples are ready for concentration.

8.3 *Concentration*

8.3.1 Rinse all concentrating glassware with DCM.

8.3.2 Label Kuderna Danish tubes with corresponding ID numbers to
sample extracts, and add a Teflon boiling stone to each tube.

8.3.3 Assemble concentrator reservoir and KD tube, ment the reproducibility of the results.

8.3.4 Place on top of hot water bath until liquid volume reaches approximately 10.0ml.

8.3.5 Solvent exchange by slowly adding 60mls of hexane to the top of the snyder column.

8.3.6 Concentrate down to 1.0ml and remove from bath. Allow all solvent to condense down.

8.3.7 Remove snyder column and KD flask. Attach micro snyder column with clip and concentrate to 1.0ml.

8.3.8 0.5 ml of the sample is put into a 2.0 ml vial with a teflon cap and stored in the sample fridge. The other 0.5 ml of the sample is combined with 0.5 ml of the fractionation surrogate in the KD flask.

8.3.9 Samples are ready for fractionation.

9.0 Fractionation

9.1 Set up Sep-Pak manifold with liners and stopcocks.

9.2 Place silica Sep-Paks on top of the stopcock making sure the screw valves are loose so flow is not restricted.

9.3 Rinse Sep-Paks with 30 mls of Hexane, stopping when solvent reaches top of frit.

9.4 Load 1 ml of sample (0.5 ml of fractionation surrogate combined with 0.5 ml of sample) into the Sep-Pak. Rinse KD tube with 1 ml fresh Hexane and add to top of Sep-Pak, repeat. Then rinse sample through with 19 mls of Hexane. Do this by adding 5 mls, 4 mls, and then 10 mls each time allowing enough time for the sample plug to move down the Sep-Pak. Collect sample in a KD tube or vial with teflon cap. This is the Aliphatics fraction.

9.5 Remove samples from manifold and replace collection tube with new/clean ones.
9.6 Rinse Sep-Pak with 6mls of DCM followed by 6 mls and then 10 mls each time allowing the plug to move down and the solvent to drain.

9.7 Collect sample in KD tube or vial with teflon cap. This is the Aromatics fraction.

9.8 Attach KD flask and synder column a place on bath. Do this with both the aliphatics and the aromatics fractions.

9.9 Concentrate both the aliphatics and aromatics fractions down to 1.0mls.

9.10 The aliphatics portion goes to the GC/FID lab and the aromatics portion is split in half, one half goes to the GC/FID lab and the other goes to the GC/MSD lab where they are analyzed.

10.0 **GC Conditions**

10.1 Oven program: Set oven temperature to 60 C for 3 minutes, then 9 C/min to 300 C and hold for 12.0 minutes. Total run time is 41.67 minutes.

10.2 Autosampler injection is 1uL.

10.3 FID temperature, 315 C

10.4 Injection port temperature, 285 C

10.5 Column head pressure 16.0psi at 60 C.

10.6 Column from Restek: RTX-5 30m long x 0.32mm I.D. x 0.25um film thickness.

11.0 **Calculations**

11.1 External standard calibration

Concentration for soils (mg/kg)=\frac{(Ax)(A)(Vt)(D)}{(As)(Vt)(Wd)}

Concentration for liquids (ug/L)=\frac{(Ax)(A)(Vt)(D)}{(As)(Vt)(Vs)}

where:

Ax= Response for sample (analyte) in area counts
A = Amt. of standard injected, uL

As = Response for external standard in area counts

Vi = Volume of extract injected, uL

D = Dilution factor

Vt = Volume of total extract, uL

Vs = Volume of sample extracted, mL

Wd = Dry weight of sample extracted, g

11.2 Samples are calculated based on a 5 point curve.

11.3 The area of each range for each point is calculated manually. This data is entered under Edit Compounds on page 3 of QC Enviroquant. Be sure to multiply the concentration of each level by how many peaks are in that range and subtract surrogates out.

11.4 Once the levels of the curve are in go to Report Response Factors to Printer under InitCal. Make sure the RSD for each compound is less than 25% and you have linearity through the origin (0,0).

11.5 Once the initial curve is in, a CC (at 50ug/ml) must be run with every sequence, at the beginning, after every 10 samples, and at the end of the sequence. This is done to confirm that the instrument’s response and performance are acceptable. If CC parameters are not met, check instrument for problems and re-run.

11.6 The CC is calculated choosing Calculate and Generate Report under Quant. This allows the area of the ranges and the individual compounds to be calculated by the computer. The area of all the compounds is then calculated manually for each range and entered in Edit Compounds under CC area.

11.7 For each sample the analyst must calculate the surrogate and spike (if it’s a matrix spike sample or an LCS) recoveries and the Multiplier. The default surrogate concentration is 4000ug/kg for soils and 40ug/L for waters. The default spike concentration is 5000ug/kg for soils and 50ug/L for waters.

11.8 Surrogate concentration (mg/kg)= (surr amt X surr conc)/sample amt/TS

surr amt = amount of surrogate added to the sample (ml)
surr conc = concentration of the surrogate injected into the samples (ug/ml)

sample amt = amount of sample used (kg or L)

TS = total solids

11.9 Multiplier = conc vol/sample amt/TS X DF

conc vol = volume the sample was concentrated to (ml)

sample amt = amount of sample used (kg or L)

TS = total solids

DF = dilution factor

12.0 Quality Control

12.1 The Quality Assurance officer and the laboratory Director must be informed of any changes in procedure not described in this SOP.

12.2 The RPD for the sample and the duplicate must be < 50% to pass quality control.

12.3 The recovery of the surrogates and spikes must be between 40% and 140% to pass quality control.

12.4 The surrogate and spike recoveries for the LCS are recorded on control charts daily. This is done to ensure lab performance and efficiency.

13.0 References


13.2 Method for the Determination of Extractable Petroleum Hydrocarbons, Massachusetts Department of Environmental Protection.
MEMORANDUM

TO:        Gary Gonyea, MassDEP
           Melissa Provencher, BRPC
           
FROM:      David C. Nyman, P.E.
           Windsor & Cady Brooks Watershed Study – Amendment to
           QAPP
           
SUBJECT:   Windsor & Cady Brooks Watershed Study – Amendment to
           QAPP
           
JOB NUMBER: 186-13

DATE:      August 26, 2010

This memorandum documents a modification of the QAPP for the Windsor & Cady Brooks Sediment and Turbidity Management Study. Specifically, the automated collection of turbidity data at the two aqueduct intake structures will be suspended at the end of August, 2010. Instead, historic data for the period of January through December 2009, consisting of once-daily observations of precipitation and influent turbidity, as available from water department archives, will be examined to document the relationship between rainfall events and turbidity observations at the intake to the Cleveland Reservoir Treatment Plant (e.g., typical time lag between storm events and elevated turbidity levels, approximate times for elevated levels to return to base-line conditions, or other trends that might be indicated by the data.).

Turbidity monitoring of Windsor (East Branch) and Cady Brooks is done by automatic sensing/recording equipment installed at the two aqueduct intake structures. Monitoring began in early June. Sixteen daily rainfall events varying from approximately 0.1 inch to 1.1 inches have been recorded at the water plant during the sampling period through the end of July, and several more are anticipated in the August data set. This data should be sufficient to qualitatively characterize the turbidity response of each brook to rainfall events, and to compare the relative turbidity response time and levels between the two brooks.

To date, no storm event has resulted in the water department personnel closing the aqueduct intake structures to prevent a high turbidity occurrence at the Cleveland Reservoir water treatment plant influent. From our discussions with water department staff over the course of this study, such conditions occur infrequently. Based on this information, we do not believe that leaving the monitors in place on the remote chance of a tropical storm is a prudent use of the resources assigned to this project.

Instead, the study team will review a selected set of historic data for the Cleveland Reservoir treatment plant influent, to further our understanding of the reservoir response to rainfall events. We will review the record of once-daily observations of rainfall and influent turbidity at the treatment plant for January through December 2009. We understand this period encompasses a time with examples of relatively higher recorded levels of turbidity encountered at the reservoir intake under current operating practices. This information will help us better understand how this water collection and storage system is affected by rainfall-induced turbidity conditions.
MEMORANDUM

The review of the historic data will consist of the following:

1. Obtain record of daily turbidity readings and daily log of precipitation measured at the treatment plant. This data is available in printed format, not in digital format.
2. Transcribe the data to Excel spreadsheet format and plot turbidity versus date and rainfall versus date for the period of record.
3. Review the data for qualitative relationship between occurrence of rainfall events and occurrence of elevated levels of turbidity at the water plant reservoir intake structure. For example:
   a. Are most precipitation events typically followed by a rise in turbidity level in the reservoir?
   b. Do greater amounts of precipitation result in relatively greater turbidity readings?
   c. Is there a discernible time lag between rainfall events and elevated turbidity levels measured at the reservoir?

Data quality will be addressed as follows:

1. The daily measurement log furnished by the treatment plant will be transcribed to digital format by one member of CEI staff, and a second staff member will review the Excel data for consistency with the data furnished by the treatment plant.
2. Turbidity data collected at the treatment plant is subject to the protocols for instrument calibration and data collection established for the operation of the treatment plant under current state regulations.
3. Rainfall data from the treatment plant will be compared for reasonableness with daily rainfall totals at the Pittsfield Airport, using digital files available on-line through Weather Underground. Such comparisons will be approximate, given the natural variability of precipitation events across the local geography.

The QAPP modification is essentially equivalent to the approved QAPP in meeting approved objectives. The modification only shortens the data collection period (by about three weeks); data has been obtained to date for multiple precipitation events and can be used to meet the original objectives to characterize:
- how turbidity/flow in each stream responds to the precipitation events,
- how turbidity levels differ between the two brooks over this period, and
- how changes in daily reservoir turbidity levels correspond to these events.

The modification allows the study team to supplement the knowledge gained from the program to date, by examining additional data specific to the reservoir.